***United States Court of Appeals***

***for the Second Circuit***

# APPENDIX

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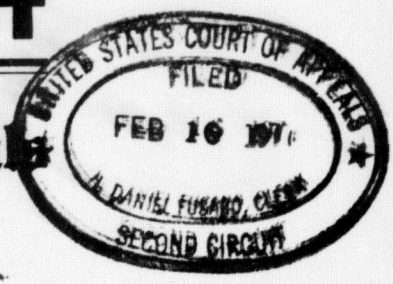
ORIGINAL




## United States Court of Appeals
For the Second Circuit

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EUTECTIC CORPORATION, NEW METALS CORPORATION and METALLIZING COMPANY OF AMERICA, INC.,

*Plaintiffs-Appellees and Cross-Appellants,*

*v.*

METCO, INC.,

*Defendant-Appellant and Cross-Appellee.*

B
8/5

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## JOINT APPENDIX

**VOLUME VIII OF EIGHT VOLUMES**

**(EXHIBITS — Pages E946 to E1233)**

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The Court is respectfully requested to disregard the underlinings, marginal notations, asterisks and other markings of plaintiffs' counsel appearing on the pages reproduced in the Joint Appendix.

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PX-24

1(1) Ni Al (powder - prep by SB, pinhead size)
2(2) " " (Tis Baeza)
3(3) " " + $Al_2O_3$
4(4) " " + " " (and $ZrO_2$, tablets)
5(11) Ni O "
6(20) Ni Ti " * (Wire gauze art)
7(25) Cu Al " ( " " " ; <750 mesh)
8(30) Ni Al wire, product from Al-Ni powder
9(32) " " - cast ingot (e.g. 2 in. dia)
10(33) " " - wire, 5 mil
11(43) Ni Si powder
12(44) Si Mo "
13(49) Si Ti "
14(50) Al Dy " * ①
15(51) Al La " * ①
16(52) Al Ce "
17(53) Ce Al "
18(?) Ni Al powder + metals

* Best if made economical by spraying
① Might use higher rare earth content which would improve oxidation resistance

E 947

1) Na Al vers — Al en Na Al mixture (sulfurique)
2) " " — " " Na Al mixture "
3) Ca Al " — Al en Ca Al mixture "
4) Ca Al " — " " Ca " "
5) Ta Al " — " Ta " "
6) B Al " — " B " (ignition
7) Cl Al (ex. en Al 5,5 % solution) — Al en poudre mixture "
8) Na Al (ex. B. C solution) — " —
9) " ( " Al ? " ) — " —

E 948

# Exothermics & Possible Exothermics.

Possibly exothermic. Col 3 not investigated bu
compounds form

PX-25

| | | (1) Metals Exo's | Metals over 2550°F | Metals lower MP (2) | (3) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ag | Silver | 5 | | | 5 | 17 | | | | | | | | | |
| Al | Aluminum | 30 | 1 | 1 | 31 | 8 | * | | | | | | | | |
| As | Arsenic | 7 | 2 | 23 | 30 | 10 | | | | | | | | | |
| Au | Gold | 7 | 2 | 8 | 15 | 13 | | | | | | | | | |
| B | Boron | 30 | | 9 | 39 | | ? | | | | | | | | |
| Ba | Barium | 7 | | 9 | 16 | 4 | | | | | | | | | |
| Bi | Bismuth | 11 | | 9 | 20 | 11 | | | | | | | | | |
| Be | Beryllium | 8 | 13 | 14 | 22 | 6 | | | | | | | | | |
| C | Carbon | 3 | | 11 | 14 | 10 | | (1) | From "Colu | | | | | | |
| Ce | Cerium RE | 12 | 1 | 10 | 22 | | | (2) | " | " | " | | | | |
| | Calcium | 7 | 1 | 12 | 19 | 10 | | (3) | " | | | | | | |
| Co | Cobalt | 4 | 7 | 10 | 14 | 5 | | | | | | | | | |
| Cr | Chromium | 4 | 6 | 12 | 16 | 12 | | | | | | | | | |
| | Cadmium | 6 | | 15 | 21 | 2 | | | | | | | | | |
| | Casium | 1 | | 4 | 5 | 6 | | | | | | | | | |
| | Cerium RE | 1 | | 4 | 4 | 25 | | | | | | | | | |
| Fe | Iron | | 8 | 9 | 9 | 22 | | | | | | | | | |
| Gd | Gadolenium | 1 | | 4 | 5 | 2 | | | | | | | | | |
| Ge | Germanium | 8 | | 8 | 16 | 10 | | | | | | | | | |
| | | 3 | 2 | 22 | 25 | 10 | | | | | | | | | |
| H | Hydrogen | 1 | | | 1 | | | | | | | | | | |
| Hg | Mercury | 7 | | 11 | | 13 | | | | | | | | | |
| Hf | Hafnium | 5 | | 5 | 10 | 5 | | | | | | | | | |
| In | Indium | 5 | | 9 | 14 | 9 | | | | | | | | | |
| Ir | Iridium | 1 | | 6 | 7 | 3 | | | | | | | | | |

OK to O.

21
1
19
2
24
67

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ~~Ga~~ | ~~Gallium~~ | 7 | | | | | | | | | | | |
| K | Potassium | 7 | | 3 | 10 | 3 | | | | | | | |
| La | Lanthanum RE | 11 | 1 | 7 | 18 | 5 | | | | | | | |
| Li | Lithium | 11 | | 3 | 14 | 2 | | | | | | | |
| Mo | Molybdenum | 2 | 6 | 6 | 8 | 5 | | | | | | | |
| Mg | Magnesium | 10 | | 9 | 19 | 17 | | | | | | | |
| Mn | Manganese | 4 | ~~85~~ | 4 | 8 | 22 | | | | | | | |
| | ~~Niobium~~ Columbium | 5 | 6 | 12 | 17 | 4 | | | | | | | |
| Nd | Neodymium RE | 3 | | 3 | 6 | | | | | | | | |
| Ni | Nickel | 6 | 3 | 8 | 14 | 21 | | | | | | | |
| Np | Neptunium | 2 | | 2 | 4 | | | | | | | | |
| N | Nitrogen | 4 | | 26 | 30 | | | X | | | | | |
| Na | Sodium | 10 | | 4 | 14 | 5 | | | | | | | |
| O | | 17 | | | 17 | | | X | | | | | |
| Os | Osmium Pt | 2 | | 14 | 16 | | | | | | | | |
| Pr | Prasodymium RE | 8 | | 3 | 11 | 3 | | | | | | | |
| P | Phosphorous | 2 | | 29 | 31 | 8 | | | | | | | |
| | Protactinium | | | | | | | X | | | | | |
| Pd | Palladium Pt | 2 | 2 | 11 | 13 | 12 | | | | | | | |
| | Polonium | | | | | | | X | | | | | |
| Pu | Plutonium | 6 | | 15 | 21 | 3 | | | | | | | |
| Pt | Platinum Pt | 2 | 4 | 20 | 22 | 10 | | | | | | | |
| Pb | Lead | 12 | | 4 | 16 | 10 | | | | | | | |
| | | | | | | | | Re | Rhenium | | | | |
| | | | | | | | | Ru | Ruthidium | | | | |
| Ru | Ruthenium Pt | 2 | 1 | 18 | 20 | 3 | | Rh | Rhodium | X | | | |
| S | Sulfur | 4 | | 24 | 28 | 16 | | | | | | | |
| Sb | Antimony | 10 | | 10 | 20 | 15 | | | | | | | |
| | | | | | | | | X | | | | | |
| Se | Selenium | 8 | 1 | 21 | 29 | 10 | | | | | | | |
| Si | Silicon | 6 | 8 | 20 | 26 | 14 | | | | | | | |

E 951

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| ~~Si~~ | ~~Silicon~~ | ~~6~~ | ~~8~~ | ~~20~~ | ~~26~~ | | | | | | | | |
| ~~Sn~~ | ~~Tin~~ | ~~13~~ | ~~2~~ | ~~10~~ | ~~23~~ | | | | | | | | |
| Sr | Strontium | 3 | | 11 | 14 | 2 | | | | | | | |
| Sn | Tin | 13 | 2 | 10 | 23 | 13 | | | | | | | |
| ~~S~~ | ~~Sulfur~~ | ~~4~~ | | ~~21~~ | ~~25~~ | ~~16~~ | | | | | | | |
| Te | Technetium | | | 1 | | | | | | | | | |
| Te | Tellurium | 12 | | 31 | 43 | 8 | | | | | | | |
| Th | Thorium | 7 | | 13 | 20 | 4 | | | | | | | |
| Ta | Tantalum | 4 | 5 | 14 | 18 | 4 | | | | | | | |
| Ti | Titanium | 5 | 6 | 14 | 19 | 11 | | | | | | | |
| Tl | Thallium | 9 | | 5 | 14 | 3 | | | | | | | |
| U | Uranium | 8 | | 18 | 26 | 8 | | | | | | | |
| V | Vanadium | 5 | 2 | 14 | 19 | 6 | | | | | | | |
| W | Tungsten | 3 | 6 | 8 | 11 | 9 | | | | | | | |
| Y | Yttrium | 1 | | | 1 | 2 | | | | | | | |
| Yb | Ytterbium RE | 1 | | 2 | 3 | 1 | | | | | | | |
| Zn | Zinc | 5 | | 9 | 14 | 12 | | | | | | | |
| Zr | Zirconium | 10 | 11 | 12 | 22 | 10 | | | | | | | |
| ~~Fe~~ | ~~Iron~~ | | ~~8~~ | ~~9~~ | ~~9~~ | ~~22~~ | | | | | | | |
| Re | ✓ | | 3 | 9 | 9 | 4 | | | | | | | |
| ~~Cu~~ | | | | ~~14~~ | ~~11~~ | ~~25~~ | | | | | | | |
| Ga | | 1 | | 8 | 9 | 10 | | | | | | | |
| ~~Pa~~ | | | | 1 | | 1 | | | | | | | |
| Po | | | | 1 | | | | | | | | | |
| Rb | ✓ | | | 4 | 4 | 2 | | | | | | | |
| Rh | ✓ | | | 20 | 20 | 3 | | | | | | | |
| ~~Sc~~ | | | | ~~2~~ | | | | | | | | | |

E 952

| | | |
|---|---|---|
| Ag - 17 | K - 3 | Sb - 15 |
| Al - 8 | La - 5 | Se - 10 |
| As - 10 | Li - 2 | Si - 16 |
| Au - 13 | Mg - 17 | Sm - 1 |
| Ba - 4 | Mi - 1 | Sn - 13 |
| Be - 6 | Mo - 5 | Sr - 2 |
| Bi - 11 | Mn - 22 | Te - 8 |
| C - 10 | Na - 5 | Tl - 3 |
| Ca - 10 | ~~N - 4~~ | Ta - 4 |
| Cd - 2 | Nb - 4 | Ti - 11 |
| Co - | Ni - 21 | Th - 4 |
| ~~Cl - 1~~ | ~~O - 5~~ | U - 8 |
| Cr - 12 | P - 8 | V - 6 |
| Cs - 6 | Pa - 1 | W - 9 |
| Cu - 25 | Pb - 10 | Y - 2 |
| Fe - 22 | Pd - 12 | Yb - 1 |
| Ga - 10 | Pr - 3 | Zr - 10 |
| Gd - 2 | Pt - 10 | Zn - 12 |
| Ge - 10 | Pu - 3 | |
| ~~H - 22~~ | Rb - 2 | |
| Hf - 5 | Re - 4 | |
| Hg - 13 | Rh - 3 | |
| In - 9 | Ru - 3 | |
| Ir - 3 | S - 16 | |

E 953

PX-33

21 Dec 1955

DPT.33.01.03

Bud.

After you read the attached, I would like to talk to you about this powder. I really believe this material has many possibilities, but requires additional work.

In the work we did with and for Cmdr Holy, Norman Double was, I believe, attempting to form nickel aluminide coatings on molybdenum with his layered coatings of Ni and Al.

The following ideas and properties are, I think, completely new and useful:

(1) Starting with a composite powder, using the heat of the flame to effect alloying
(2) Using the heat generated by an exothermic combustion reaction to reinforce the particles in the spray stream.
(3) A self bonding oxidation resistant material both for primary coatings to protect base materials from oxidation, and use as an undercoat-oxidation-resistant bonding

E 954

# SELF BONDING INTERMETALLIC COATINGS FOR HIGH TEMPERATURE OXIDATION RESISTANCE

The high temperature strength and oxidation resistance of the intermetallic compounds, nickel aluminide in particular, are well known. Its comparatively high strength and excellent oxidation resistence at temperatures over 2000°F make it an attractive material for turbine blading and like applications.

It has long been felt that impervious coatings of nickel aluminide, fused to miscellaneous base materials, would provide excellent composite structures useful at temperatures nearly up to the melting point of the nickel aluminide (3000°F), in a variety of corrosive environments.

The first attempts to produce a fusion bonded nickel aluminide coating were made in October, 1954, using simple mixtures of nickel and aluminum powder sprayed with a powder gun and heat treated after deposition on the base material. Results with this method were not completely successful, quite inconsistent, due probably to the oxides introduced into the coating during the spraying operation. Probably for the same reason, layered coatings of nickel and aluminum applied with the wire gun and subsequently heat treated were not successful. No attempts were made to effect the formation of nickel aluminide and bond the layer to the base in an atmosphere furnace with either powder or wire spray.

When composite powders became available, I had Sherritt Gordon Mines, Ltd., prepare a special powder, the particles of which were composed of a core of aluminum overcoated with a sheath of nickel, which analized 81.5% Ni and 18.5% Al. I thought that the heat of formation of the nickel aluminide (an exothermic combination reaction) would aid in giving a self-bonding oxidation resistant coating of nickel aluminide.

(1) Even this first composition powder is self bonding on a fine ground CRS panel with only a mild preheat. So far not quite 100% bonding coverage has been achieved, but the bond strength is high enough to anchor a .050" thick #101 coating sprayed hot. Coatings of nickel aluminide separated from the base leave almost 100% of the base coated with a bonded layer of nickel aluminide. The nature of the bond and the bond strength are unknown at this time; work must be done to determine the nature of the bond, bond strengths, and the optimum Ni:Al ratio.

(2) Self-bonded coatings will fuse 100% to the base material and apparently will protect a mild steel base material from high temperature heat corrosion for extended periods of time. Long time heat corrosion tests should be run.

Self Bonding Intermetallic Coatings

(3) The formation of a nickel-aluminum compound (nickel aluminide) in the flame is positive. Post heating of an applied coating shows no bleeding of aluminum at that metal's M. P. At some temperature around 2400°F (judging by color), approximately the melting point of nickel, a few balls of bright metal exuded from pores, so I would assume a slight excess of nickel, not combined with aluminum. At the temperature at which iron oxide melts, approximately 2800°F, the coating was still intact on the face and was not melted. The M. P. of nickel aluminide has been reported as 3000°F. Oxidation had crept in from the edges, but the center face coating of the panel was intact. There were some signs of melting of the CRS base.

(4) In spite of the apparent "softness" of the coating as measured on the Rockwell Hardness Tester, the particles in the sprayed surface are hard and abrasive enough to abrade hardened steel. The literature indicated that bodies of nickel aluminide, in spite of their apparent "softness," are difficult to machine because of excessive tool wear and had to be ground.

(5) Used as an undercoat for ceramic coatings, either on a ground or grit blasted surface, the ni-al will protect the base from oxidation at high temperatures. This must also be checked for protection of molybdenum and columbium at high temperatures.

(6) As the metallic phase of a cermet containing aluminum oxide, 40% Ni-Al + 60% 101, the nickel aluminide will permit usage up to almost 3000°F. A coating approximately .010" thick withstood a number of quenches from over 2000°F to cold water. More thermal shock resistance can be obtained by increasing the nickel-aluminide : $Al_2O_3$ ratio. This 40:60 mix of Ni-Al $Al_2O_3$ did not smoke when sprayed with acetylene in the P gun.

The cermet in the one composition checked may or may not be self bonding. It is not a good bonding coat for subsequent ceramic coatings because of the extremely smooth surface of the cermet as sprayed.

By F. J. Dittrick

December 18, 1959

WITNESSED _____

DATE _____

E 956

Reprinted from The American Ceramic Society Bulletin, Vol. 41, No. 6, June 7, 1962
Copyright 1962 by The American Ceramic Society

# New Flame Spray Technique for Forming Nickel Aluminide—Ceramic Systems

PX-40

F. J. DITTRICH

Metco Inc., Westbury, Long Island, N. Y.

A flame spray process and material* has been developed for depositing nickel aluminide coatings which are "self-bonding" to the smooth clean surfaces of many substrate materials, to provide adhesion for other coatings such as refractory oxides and carbides. In addition, the new material has been used in mixture with ceramic powders to deposit cermet coatings and to build gradated cermet coating systems. The key feature of the new process is the starting material. This is a powder of composite particles, each of an aluminum core clad with a nickel sheath. The exothermic combination reaction of these metals in the heating zone of a flame spray gun results in the formation of nickel aluminide and the generation of considerable heat. A comparison is made between nickel aluminide and 80:20 nickel-chromium alloy powder when used as a bond coat for zirconia coatings, and in mixture with zirconia to form cermet coating systems. Coated panels were exposed to the high temperature flame of a plasma gun in steady-state and cyclic tests, which included radical thermal shock.

FLAME SPRAYED materials in general use today are conventional forms of metals, alloys, or ceramics, which include oxides, carbides, nitrides, borides, beryllides, or other intermetallic compounds. The material in a resultant coating is basically unchanged in chemical form from the starting material. Physical structure is somewhat different. Metal coatings contain a small amount of oxide. All coatings are porous to some degree, usually about 5 to 20%, depending on the material, the spray equipment, and the spray technique used. Combustion flame equipment is used to spray wire, rod, and powdered materials with melting points up to around 5000°F. Plasma flame equipment is used to spray any powdered material that will melt without decomposing or vaporizing on heating, no matter how high its melting point.

## Bonding Flame Sprayed Coatings

Ordinarily, grit blasting or grit blasting plus undercoating techniques are used to bond flame sprayed metal, ceramic, or cermet coatings to the substrate surface. This is required because, except for molybdenum, none of the commonly used materials is self-bonding to a smooth surface on any of the usual base materials.

Under special spray conditions such as complete atmosphere control in a chamber, high temperature preheating of the substrate, or both, other flame sprayed materials can be made to self-bond to some substrates. The rough grit blasting usually required to hold ordinary coatings with sufficient bond strength to be of practical use can deform thin-walled structures and destroy their usefulness. The bond of ceramic coatings to grit blasted materials with high thermal expansion is weak unless additional bond coatings are provided.

The radical differences in thermal expansion rates between ceramic coatings and the usual metallic base materials (such as steel) results in excess shear stress at the interface when the composite structure is temperature cycled. The stress can destroy the bond completely, even when the sprayed article is first cooled to room temperature after spraying, if the preheat and spray temperature ranges are not properly controlled. Some additional bond strength can be provided by spraying a layer of an oxidation resistant material, such as an 80:20 nickel-chromium alloy, which mechanically bonds well to grit blasted surfaces and to which the ceramic overcoats will strongly bond. This layer does not eliminate the need for as rough a grit-blasted preparation as can be provided.

Grit blasting for roughness can be eliminated by using a special "self-bonding" sprayed layer to provide the bond between the desired coating material and the substrate. Such a coating material requires only that the base be clean of all surface contamination and heavy oxide films in order to form a metallurgical bond with the substrate material. Welding of the sprayed particles occurs on impact with the base, and requires no subsequent heat treatment to effect the bond or to improve the bond strength.

Molybdenum, the most common self-bonding bond-coat material, is usually sprayed with a wire gun. It has found wide usage in applications where high temperatures in oxidizing atmospheres are not involved. Its use is restricted under these conditions, where the coating-substrate interface tem-

Based on a paper presented at the Sixty-Fifth Annual Meeting of The American Ceramic Society in Pittsburgh, May 1, 1963 (Ceramic-Metal Systems Division, No. 17-R-63).

* Patent pending.

## The Author

Ferdinand J. Dittrich

Ferdinand J. Dittrich has been associated with the metallizing and flame spray process for over 26 years. Except for his service during World War II, he has been with Metco Inc. since 1938. At first he worked as a technician and draftsman, while he studied engineering at night. He has held engineering positions with the company since 1945 and is presently senior engineer in the Research and Development Department. A number of patents have been issued to him and others are pending, all relating to flame spray equipment, processes, and materials.

Mr. Dittrich is the inventor of the composite nickel-aluminum powder for the flame spray process. His original ideas and studies with exothermically reacting materials began over 10 years ago. The actual development of a practical composite flame spray powder had to wait for the relatively new technology of cladding powders to become available, but once this occurred the new type of powder was possible.

perature exceeds approximately 600°F, because of its high oxidation rate and the volatility of its oxides at somewhat higher temperatures. A number of other materials, such as tantalum and columbium are self-bonding to some substrates under normal spray conditions, but are not widely used because of their high cost. Actually nearly any metal including tungsten or even steel can be self-bonding to some substrate materials when sprayed under conditions of complete atmosphere control in a chamber, or when the substrate is heated to an elevated temperature, or a combination of the two. However, this is rarely practical. Oxide ceramics are generally more difficult to bond to smooth surfaces.

### Nickel Aluminides

Early studies of the nickel aluminides in 1933 by Maxwell and Gruse[1] indicated that these compounds possessed excellent oxidation resistance, high strength, and good impact strength. These attributes are primary requirements for a material which is to be used as a bond coat at high temperatures in oxidizing atmospheres. The formation of the nickel aluminides from the chemical combination of nickel and aluminum is accompanied by the release of considerable heat. For example, when a mixture of nickel and aluminum powders is heated to over the melting point of the aluminum, there is a violent exothermic combination reaction. There are a number of nickel aluminide intermetallics formed, NiAl having the highest melting point, reported at 2881°F.[2] This is substantially higher than either nickel or aluminum.

### Flame Sprayed Nickel Aluminide

This material may be flame sprayed in the usual manner starting with preformed nickel aluminide powder. The deposited coating is not self-bonding and has the typical porous structure of most sprayed coatings. Flame sprayed mixtures of nickel and aluminum powders do not react in the flame to form the nickel aluminides and the coating produced is simply a mixture of nickel and aluminum particles.

The starting powder for the new process consists of composite particles, each of which has a core of aluminum [illegible] rounded by a shell of nickel. This form of nickel aluminum powder allows initiation of the exothermic combination reaction [illegible] the formation of the intermetallics

takes place, and the generation of considerable heat. It produces coatings of nickel aluminide which are self-bonding, more coherent, and less porous than the usual flame sprayed coatings.

During spraying, evidence of the exothermic reaction is apparent from the extra-bright particles in the spraystream. Motion pictures taken at 8000 frames per second showed the individual particles glowing more brightly as their temperature increased as they traveled further from the nozzle, away from the hottest zone of the combustion flame. This is in contrast to spraying other powders where the particle temperature reaches its peak in the hottest part of the flame and then decreases during travel away from the nozzle. While the term "ignition" is used to describe the initiation of the reaction in the flame it should be understood that no combustion is involved; the combination reaction does not require and is not supported by oxygen. This reaction will proceed to completion in vacuum or under inert or reducing gas cover. However, during the spraying in air, there is some reaction of the hot particles with atmospheric oxygen which produces some smoke and oxide, but this is independent of the combination reaction.

Using special sample preparation procedures and an optical metallograph, Longo[3] revealed alloy layers at the interface between nickel aluminide particles and a hardened 1095 steel substrate. Figure 1 shows a photomicrograph of such a layer. Longo chose the hardened steel to prevent relief from the nickel aluminide during polishing. Prior to coating, the substrate was alternately polished and etched to remove any disturbed metal and finally polished to approximately 4 microinch (aa) finish. The actual average thickness of this layer is about 0.1μ. The darkened area is the martensitic structure of the steel substrate etched with Nital; the light area is an unetched particle of nickel aluminide. The narrow zone between is the alloy bond layer. While the composition of this layer has not yet been determined, it is expected that this will prove to be an iron-nickel-aluminum alloy.

### Ceramic and Cermet Coating Systems

This new powder has proved effective in bonding metal, ceramic, cermet, and gradated cermet coating systems to various substrate structures. As noted before, grit blasting for roughness for bonding or grit blasting plus intermediate coating layers for improving the bond, can be eliminated by using this self-bonding material. In addition, an improvement in bond strength is noted. Tensile tests showed that the bond strength of zirconia coatings with a 90:20 nickel chromium alloy undercoat on a grit-blasted steel base is

Fig. 1. Nickel aluminide particle (upper) and a hardened steel substrate (lower) showing the alloy bond layer between them. The interface has been extended 14,000 times by tapersectioning at 4° to the substrate. (Steel etched with Nital; nickel aluminide unetched.) X1000

Fig. 2. Section through a steel base–nickel aluminide–sprayed steel coating system. (Nital etch) ×220

approximately 900–1100 psi, while with a nickel aluminide bond coat on a smooth ground steel base it is 1100–1400 psi.

Figure 2 is a section through a coating system in which the surface-ground steel substrate was sprayed with a bond coat and then overcoated by spraying with a low shrink alloy steel. Note particularly the straight line near the bottom of the picture which is the interface between the substrate and the bond coat, and the overhanging particles on the surface of the bond coat into which the overcoat has "keyed."

The tensile strength test[6] used consists of spraying a bond-coat on the smooth-machined face of a 1 in. diameter cylinder of the desired substrate material. An overcoat material is then sprayed onto the bond coat and is ground smooth and flat. After the face of a mating cylinder is cemented to the ground overcoat surface, the assembled structure is stressed to failure in tension. The ultimate tensile strength of the coating system is then calculated. Visual examination of the break indicates where in the coating system the failure occurred.

Failure in the coating system of zirconia bonded with nickel aluminide occurs either in the ceramic layer or at the ceramic-bond coat interface, or in both places, indicating that the tensile strength of the ceramic generally is lower than that of any other component in the system.

In the system of a metal coating bonded with nickel aluminide, illustrated in Fig. 2, failure in the tensile strength test[6] generally occurs in the bond coat layer, indicating that the tensile strength of the system is the strength of the bond coat layer when stressed in the direction parallel to the direction of spray. Because the failures occur as they do, we have never been able to determine the actual strength of the welded particle-to-substrate bond. The tensile strength range, when sprayed with combustion flame equipment is from 2750 to 3200 psi. When sprayed with plasma flame equipment, tensile strength range is from 3000 to 3500 psi.

Figure 3 shows a section through a nickel aluminide–zirconia cermet coating system applied to a steel substrate with a plasma flame spray unit. The system illustrated consists of a 0.001 in. thick bond coat on the mild steel base, 0.001 in. of a 75% nickel aluminide–25% zirconia cermet, 0.005 in. of a 25% nickel aluminide–75% zirconia cermet, and an overcoat of 100% zirconia. The various layers are easily distinguished in this cross section. Graduated coating systems in which the proportions of each element can be continuously varied from 100% nickel aluminide for the bond coat to 100% zirconia for the topcoat can also be applied by using a dual powder feed system.

A number of coating system samples have been tested in various plasma flame and chemical flame environmental test facilities, and these tests are being continued. Cermet coatings consisting of approximately 40 wt% of nickel aluminide and 60 wt% of aluminum oxide on 1 by 3 by ⅛ in. steel coupons have been cycled between 1800°F and a cold water quench at least 10 times without any spalling of the coating due to thermal shock or loss of coating due to oxidation of the base. The coated panels were heated in air in the flame of an oxyacetylene burner.

## Plasma Flame Tests

High temperature steady-state and cyclic tests, which included radical thermal shock, have indicated that plasma flame sprayed nickel aluminide is superior to plasma flame sprayed 80:20 nickel-chromium alloy as a bond coat for ceramic or cermet overcoats.

Coated panels were prepared for testing in an arc-plasma materials evaluation facility, the design of which was based on investigations at the University of Dayton Research Institute, Dayton, Ohio.[7] Heat source was a gas-sheath stabilized DC arc plasma torch.

A water cooled plenum chamber was mounted at the nozzle exit, as indicated in the schematic drawing, Fig. 4. Oxygen was introduced into the nitrogen plasma stream at this point, in the proper proportion to simulate air atmosphere. Heat flux was calibrated with a cold wall calorimeter with water cooled guard ring mounted on a slide on which the test specimen was also mounted. The purpose of the calorimeter was to assure repeatable flame conditions, and was not for ab-

Fig. 3. Section through a nickel aluminide–zirconia cermet coating system

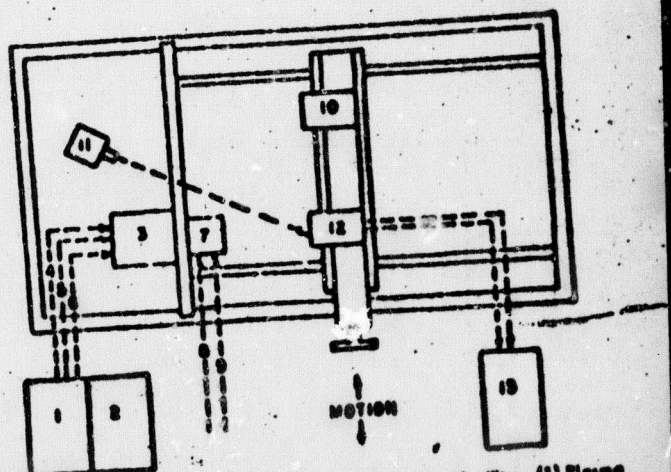


Fig. 4. Schematic diagram of plasma-arc thermal test facility. (1) Plasma control console, (2) power supply, (3) plasma-arc torch, (4) coolant for torch, (5) power for torch, (6) gas for torch, (7) water-cooled plenum chamber, (8) metered mixing gas, (9) coolant, (10) cold-wall calorimeter, (11) optical pyrometer, (12) test panel, (13) millivolt recorder for monitoring backside temperature.

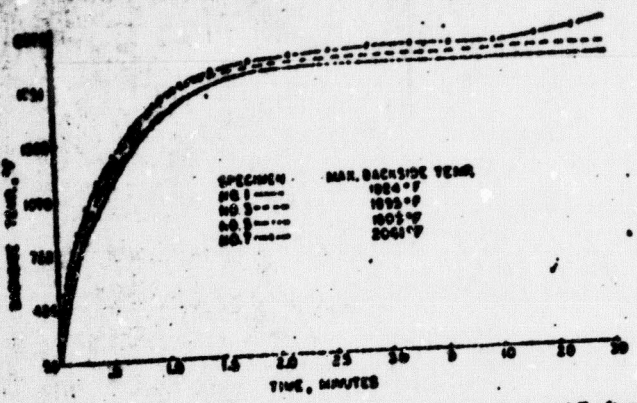


Fig. 5. Temperature curves for back-side of specimens 1, 3, 5, and 7 after heat treating for 30 minutes.

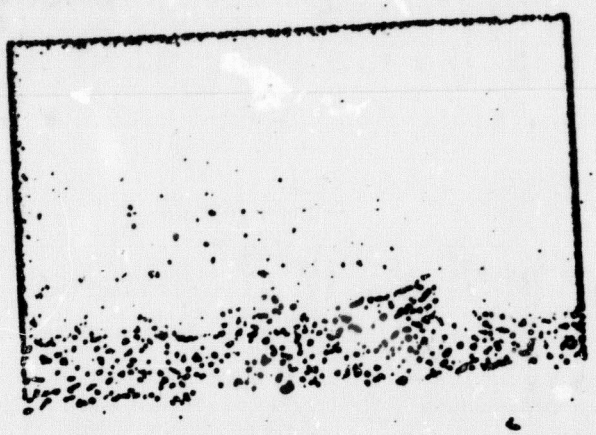

Fig. 6. Specimen 3 after 30 minutes continuous exposure to plasma flame showing no failure of nickel aluminide bond coat or zirconia topcoat. (X100)

solute heat flux values. The heat flux was adjusted to the desired value with the calorimeter in proper position; the test specimen was then moved into position for testing without shutting down the heat source. This slide also permitted removal of the specimen from the plasma stream in order to quench the specimen in the cyclic heating-cooling test procedure. The total gas environment in which the specimen was tested was not controllable; the test setup was operated in air. Specimen temperature was monitored by welding a Chromel-Alumel thermocouple to the back of the test specimen at the center of the flame impingement area. Back-side temperatures were recorded on a millivolt strip-chart recorder. Nitrogen flow was 110 SCFH, oxygen flow was 25 SCFH. Heat flux was approximately 150 Btu/ft²/sec. Power input for the plasma torch was 14.6 kw (240 amp at 61 v). The test panels were mounted at 45° to the axis of the plasma stream, 1½ in. from the nozzle face measured along the axis of the plasma stream.

Steady-state tests consisted of continued exposure to the plasma flame for 30 minutes followed by cooling in air. The thermal shock tests consisted of cycles of exposure to the plasma flame for 3 minutes followed by rapid cooling to room temperature via compressed air blasts on both front and back of the test specimen for a period of 3 minutes. The shock tests were continued for 15 cycles, or to failure. Back-side temperatures were continuously recorded for all specimens during the entire test.

Test panels were prepared by plasma-flame spraying appropriate coatings onto 2 by 4 by ⅛ in. thick cold finished mild steel panels. Panels for nickel-chromium alloy systems were grit-blasted on both sides with a 50-50 mixture of G25 and G10 steel grit at 30 psi air pressure in a suction feed blast machine. Panels for the nickel aluminide systems were grit blasted in the same manner on the back-side only and cleaned on the face to be coated by blasting with −325 aluminum oxide powder at 30 psi air pressure. Since the nickel aluminide is self-bonding, the rough, grit-blasted surface preparation required for nickel-chromium alloy was unnecessary. All panels were coated with 0.005 in. of nickel-chromium alloy on the back to retard oxidation of that surface and to equalize radiation and other heat transfer properties.

The test coatings were plasma flame sprayed according to Table I. The metal-ceramic mixtures (cermets) indicated were proportioned to result in approximately equal volume proportions of nickel-chromium alloy to zirconia, or nickel aluminide to zirconia.

Specimen numbers 1, 3, 5, and 7 were continuously exposed to the plasma flame for 30 minutes, after which they were cooled to room temperature normally, i.e., without forced cooling. Figure 5 shows the back-side temperatures of these 4 specimens, recorded for the 30 minute heat-treating schedule. The closeness of the back-side temperature curves indicates approximately equal insulating value for all 4 coatings. The reason for the gradual back-side temperature increase on specimen #7 during the last 20 minutes of the test could not be determined. Visual examination of the four specimens showed no noticeable failure during or immediately after the test.

The specimens were cut through the center of impingement area of the plasma flame, mounted, and polished for metallographic examination. Figures 6 and 7 are cross-sections through test panels #3 and #7 respectively. As illustrated by Fig. 6, specimen #3, nickel aluminide bond coat plus zirconia

Table I.

| Specimen No. | Coating System | |
|---|---|---|
| | Bond Coat | Overcoat (4) |
| 1 & 2 | 0.005 in., #401 | 0.005 in., XP1131 + 0.005 in. XP1130 + 0.005 in. #201 |
| 3 & 4 | 0.005 in., #401 | 0.005 in. #201 |
| 5 & 6 | 0.005 in., #401 | 0.005 in., #A + 0.005 in. #B + 0.005 in. #201 |
| 7 & 8 | 0.005 in., #401 | 0.005 in. #201 |

Codes:

#201 = Nichrome (80 Ni-20 Cr)
#401 = Nickel aluminide
#401C = Nickel-chromium
#A = 70 wt% #401C + 30 wt% #201
#B = 40 wt% #401C + 60 wt% #201
XP1130 = 45 wt% #401C + 55 wt% #201
XP1131 = 25 wt% #401C + 75 wt% #201

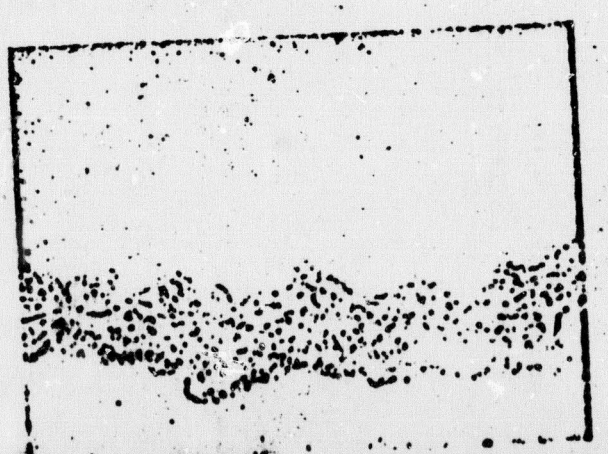

Fig. 7. Specimen 7 of nickel-chromium alloy bond coat plus zirconia topcoat. Visible failure in center of plasma impingement area after 30 minutes continuous exposure to plasma flame. (X100)

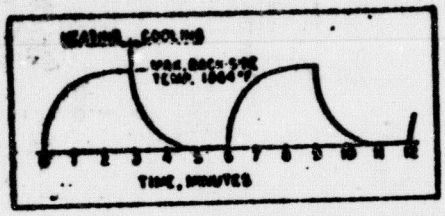


Fig. 8. Typical temperature curve for the back-side of specimens during heating and cooling cycles (specimen 4)

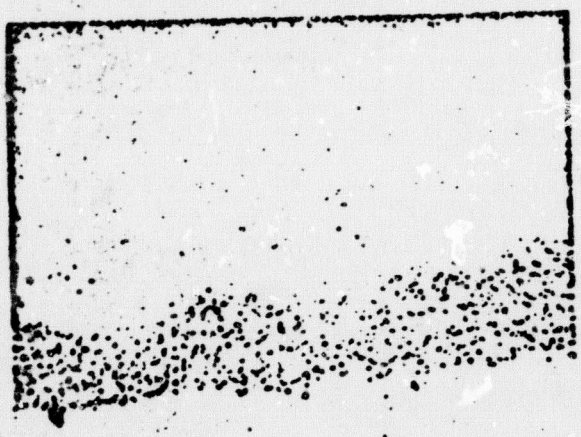

Fig. 9. Specimen 4 after 15-cycle thermal shock test showing no failure in coating system. (X100)

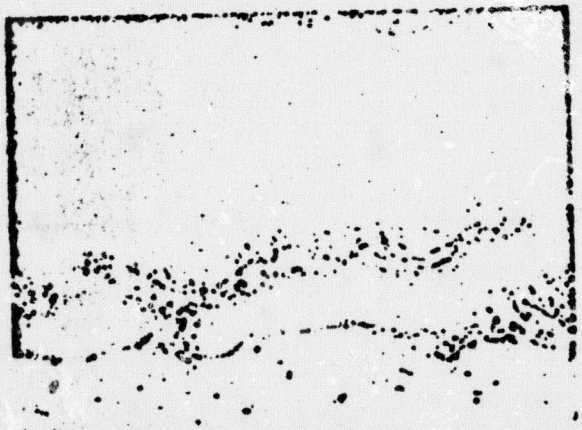

Fig. 10. Specimen 8 showing failure between substrate and nickel-chromium alloy, bond coat, and between bond coat and zirconia topcoat in the 14th cycle of thermal shock test. (X100)

topcoat, was undamaged at the end of the test. Specimen 7, nickel chromium alloy bond coat plus zirconia topcoat (Fig. 7), showed separation of the bond coat from the substrate and cracking in the zirconia near the topcoat-bond coat interface when exposed under identical conditions.

Specimens 2, 4, 6, and 8 were exposed to a maximum of 18 thermal cycles which consisted of heating for 3 minutes and cooling for 3 minutes under conditions previously described. Figure 8 shows a typical back-side temperature curve copied from the recording made during the thermal cycle test on specimen #4. In heating, panels reached maximum back-side temperature in about 2.5 minutes and then stabilized. In cooling with an air blast on both front and back faces, the back-side of the panel reached room temperature in approximately 2 minutes and remained for 1 minute, until the start of the subsequent heating cycle. Maximum back-side temperature was 1961°F.

Specimen numbers 2, 4, and 6 showed no noticeable failure during the test. Specimen #8, nickel-chromium alloy bond coat plus zirconia topcoat, failed due to separation of the bond coat from the substrate and the zirconia from the bond coat in the heating phase of the 14th cycle. Figure 9 is a cross section through the center of impingement of the plasma flame on specimen number 4 nickel aluminide bond coat plus zirconia topcoat. It illustrates that there was no failure in the coating system. Figure 10 is a similar cross section through specimen number 8, nickel-chromium alloy bond coat plus zirconia topcoat. It shows the separation of the nickel-chromium alloy from the substrate and the zirconia topcoat from the bond coat.

None of the cermet coating systems, specimens 1, 2, 5, and 6, showed any failure during or immediately after either the steady state or the thermal cycle test, so no conclusions are drawn in comparison of nickel-chromium alloy and nickel aluminide in this type of system.

## Conclusion

Study of and process research for flame-sprayed coating materials has resulted in the development of a new coating powder, nickel clad aluminum. This coating material is self-bonding to smooth surfaces of a wide variety of substrate materials and provides an excellent bond for subsequent conventional ceramic, cermet, or metal coatings. With the nickel aluminide in mixture with ceramic materials, cermet coatings and graded cermet coating systems have been tested; these show good resistance to deterioration at high temperatures, and very good thermal shock resistance. ☆

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METCO

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REPORT #102

# METALLOGRAPHY OF FLAME SPRAYED COATINGS OF METCO 404 "Nickel Aluminide" Powder

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# METALLURGY OF FLAME SPRAYED NICKEL ALUMINIDE COATINGS*

by

Frank N. Longo

**ABSTRACT:**

Coatings deposited by flame spraying a nickel/aluminum composite, either powder or wire, adhere to most substrates by fusing to form a bond layer. The heat-affected zone is minimal, less than 0.001 in. deep. During spraying nickel and aluminum combine in an exothermic reaction which facilitates bonding, and results in a coating composed of several metallic phases and oxides. Nonstoichiometric nickel rich NiAl, $Ni_3Al$ and a solid solution of aluminum in nickel are believed to form in addition to pure nickel and glassy oxides of nickel and aluminum.

# E 964

## METALLURGY OF FLAME SPRAYED NICKEL ALUMINIDE COATINGS

### INTRODUCTION:

In recent years, the flame spraying industry has witnessed a significant advance in the techniques of bonding a surface coating to a substrate material by using a sub-bond coating of nickel aluminide.

The process and materials involving the use of flame spray powder and wire in composite form were originally introduced by F. J. Dittrich.(1) (2) Today there are three self-bonding coatings, METCO 404 powder comprising a nearly spherical aluminum core with continuous nickel shell and 450 powder containing 5% aluminum clad to a nickel core and 405, a nickel-aluminum composite in wire form. Coatings can be deposited by a combustion flame spray process using oxygen and acetylene, and by plasma flame spraying. The composite METCO 450 or 404 nickel-aluminum powders or METCO 405 wire are heated to an ignition point, where the nickel and aluminum combine in an exothermic reaction to form nickel aluminide. During spraying, evidence of the exothermic reaction is apparent from the extra-bright particles in the spray stream. Motion pictures taken at 8000 frames/sec showed the individual particles glowing more brightly as their temperature increased as they traveled further from the nozzle, away from the hottest zone of the combustion flame. The particles adhere to smooth, clean metal substrates with bond strengths between 2750 psi and 3500 psi. Most other flame spray materials require surface preparation such as grit blasting. Thus, some sort of metallurgical bond is indicated.

A number of investigators have expressed interest in obtaining more information about the coating structure and the nature of the bond. This paper presents a metallurgical study of 404, 450 and 405 coatings and the bonding interface, and a discussion of the heat-affected zone in the substrate. Special sample preparation techniques which have proved useful are also described.

### EXPERIMENTAL PROCEDURE:

Initial considerations were based upon probable room temperature structures predicted from the nickel-aluminum phase diagram. (3) However, drastic deviations from equilibrium cooling rates and the inhomogeneity due to the rapid quenching during the spraying process made these results inconclusive.

It was decided to melt and cast nickel and aluminum in various proportions and use the resulting structures as guides to this study. Ten grams of each of various mixtures of powdered nickel and aluminum, and 10 g of METCO 404 powder were put in alundum crucibles and heated with an induction coil. When the temperature reached approximately 1250°F, the powders reacted exothermically and melted. The power was shut off and the melt was cooled rapidly by blowing air over its surface. Each pellet formed was sectioned

and prepared for metallographic study. These cast pellet structures, together with the structure of NiAl and Ni3Al described by Guard and Turkalo (4), were compared to the structures of sprayed coatings. Microhardness data using a Knoop indentor and a 50 g load were used to help identify the various intermetallic compounds whose hardness vary with atomic structure and aluminum content.

In addition to the metallographic study, a sample of the sprayed 404 was thoroughly ground and subjected to X-ray diffraction analysis using cobalt radiation in a typical Debye-Scherer camera with 57.5 radius.

BOND LAYER AND HEAT-AFFECTED ZONE:

Samples of steel substrates were coated by flame spraying using combustion flames and also plasma flames. Metallurgical studies on these samples to determine the nature of the interface met with several difficulties. Problems were encountered when normal sample preparation techniques were employed, i.e.

1. The nickel aluminide coating was harder than the steel substrate, making it very difficult to obtain a relief-free interface.

2. Cold working of the substrate surface during preparation (surface grinding or grit blasting) interfered with observations of the heat-affected zone.

To solve these and other problems, the following technique was used: High carbon steel (1095), quench-hardened to a fully martensitic structure, was used as the substrate. The surface selected for spraying was alternately etched (with Nital) and polished to remove all disturbed metal. The resultant structure was the normal white martensite which readily shows the effect of subsequent heat tempering.

Immediately after the final polish, a 0.010 in. thick coating of 404 nickel aluminide was applied by the combustion flame spray process. This specimen was carefully surface ground in a plane at 4 deg to the hardened substrate. The exposed surface was then mounted and prepared for metallographic investigation. The final polishing step was done with a vibromatic polisher, which effectively minimized particle pull out. Cutting through the interface at the low angle of 4 deg gives the effect of increasing the thickness of any layers approximately X14-1/2. The similar hardness of the steel base and the coating minimized relief at the interface.

Other specimens were sectioned at 90 deg to the interface for study of the heat-affected zone, and in addition, others were polished down through the coating to the interface for study in a plane parallel to the substrate. To obtain a polished plane at the interface between the sprayed coating and substrate step-by-step polishing with frequent observation was conducted.

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* Patent pending on powder and process.

A necessary step in the study was the development of proper techniques for polishing and etching the sprayed coating-substrate systems. The methods used are described in the Appendix.

METALLOGRAPHIC RESULTS:

METCO 404 Metallographic examination of sprayed coatings revealed a continuous structure of individual particles composed of several metallic phases and oxides - Fig. 1, 2 and 3.

The structure present in a cast pellet obtained by melting the nickel clad aluminum powder is shown in Fig. 4. By direct comparison with photomicrographs of similar specimens by Guard and Turako(4), the striated phase was identified as a transformed non-stoichiometric, nickel rich, nickel aluminide (stoichiometric NiAl contains 68.50% wt% Ni) while the unetched phase was identified as $Ni_3Al$.

Using these characteristic features observed in the pellets as a guide, transformed nonstoichiometric NiAl particles similar to structures found in the castings were found in sprayed coatings, as shown in Fig. 5. Other particles of similar hardness which do not show the characteristic striations may be a retained high temperature form. In addition to these particles, there are also soft unetched particles. These can be either $Ni_3Al$ or a solid solution of aluminum in nickel or, perhaps, a combination of the two. Microhardness data in Table 1 substantiates the differences between phases and closely agrees with the hardness data published by Grala(5) for NiAl and $Ni_3Al$.

According to Bradley, et.al.,(6) the color of NiAl will change from a yellow for the nickel rich NiAl through gray and blue at the stoichiometric composition. Microscopic examination of the polished but unetched surface of a sprayed coating shows a distinct color change from particle to particle and/or within a single particle. This color change was observed to exist only for those particles of high hardness or those which etch with the striated structure.

Additional tests for free aluminum in the coating were made by treating with a 5% NaOH solution. There was no reaction to indicate any aluminum in nitrogen plasma sprayed or ThermoSprayed coatings, except in rare cases, particularly if spraying was done improperly such as at the wrong spray distance. On the other hand, argon plasma sprayed 404 usually has identifiable free aluminum.

The nominal composition of each METCO 404 particle, shown in Fig. 6, is 80% Ni and 20% Al. Under equilibrium conditions this would place the alloy in the two-phase region of NiAl + $Ni_3Al$. However, in sprayed coatings the proportions vary from particle to particle. It is believed that this is primarily due to a loss of nickel and aluminum as oxides. The final composition distribution may be as much as ± 5% from the nominal, and may partially explain the reason for the structural change from particle to particle in the coating. Different quench rates among particles also contribute to structure differences.

METCO 405:

METCO 405 is an especially developed nickel-aluminum composite wire for bonding. It is chemically similar (80% nickel - 20% aluminum) to its powder equivalent METCO 404. The coatings produced, illustrated in Fig. 7, contain almost all non-stoichiometric NiAl, estimated between 80 and 90%, the remaining particles and/or portions of particles being $Ni_3Al$ or aluminum/nickel solid solution. The reason for the deposition of more hard nickel aluminides is that most of the exothermic reaction and alloying takes place at the molten wire tip just prior to atomization. A microhardness survey of those particles identified as non-stoichiometric NiAl show a range from $KHN_{50}$ 410 to 479, depending upon the percentage of aluminum in the phase. Study of Fig. 7 shows various shading in particles which is typical of color variations normal to NiAl and described by Bradley, et.al.(6) Approximately 5% of the particles have a hardness of $KHN_{50}$ 270 which closely corresponds to $Ni_3Al$ or nickel/aluminum solid solution. The coating hardness of Rc22 also reflects the large percentage of hard nickel aluminides.

METCO 450:

METCO 450 is a composite powder comprising 4.5% Aluminum clad to a nickel core. The exothermic combination results in coatings of very high density. Particle structure was compared to standards as were METCO 404 coatings and found to be primarily nickel with some nickel/aluminum solid solution. Only minor percentages of non-stoichiometric NiAl and $Ni_3Al$, estimated at less than 5%, were found. Because of the relative absence of hard nickel aluminides ThermoSpray coatings are soft, Rb 65 and plasma coatings depending upon choice of plasma gas vary from Rb 60 to Rb 80. ThermoSpray and plasma spray coatings are illustrated in Figs. 8, 9 and 10. Unetched coatings show none of the characteristic gray and blue colors indicative of non-stoichiometric NiAl. Microhardness verifies the absence of NiAl since particle hardness of ThermoSpray coatings is $KHN_{50}$ 150, and plasma coatings $KHN_{50}$ 193. Because of the small quantity of aluminum in 450 powder there is seldom any free aluminum found in a properly sprayed coating.

X-RAY DIFFRACTION STUDIES:

An X-ray diffraction analysis was conducted on 404 coatings to further identify phases and afford correlation with metallographic results. The data, although interesting was not conclusive. The major identifiable crystalline phase found was Ni. There were many unidentified lines (see Appendix) in the film which do not coincide with any of the known aluminum nickel compounds, nor to the oxides of nickel or aluminum. To further complicate the picture, Bradley(6) reported that the identifiable lines of quenched $Ni_3Al$ are scarcely different from Ni or the solid solution of aluminum in nickel. Thus, considering the rather drastic quench rate normal for flame sprayed coatings, it appears that X-ray analysis cannot distinguish between Ni, $Ni_3Al$ or the solid solution of aluminum in nickel.

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It can also be expected that X-ray analysis would not pick up small quantities of nickel rich, non-stoichiometric NiAl if contained with other phases. Typically, as much as 15% may go undetected. The National Bureau of Standards has data only on stoichiometric NiAl, and there is no standard for quenched non-stoichiometric NiAl. It is possible then that the unidentified lines in the film are for the non-stoichiometric NiAl.

OXIDES:

The black phase readily observed in all coatings of METCO 404, 450 and [illegible] is an oxide typically associated with nickel aluminide coatings.

Combustion coatings contain more oxide than the plasma coatings, presumably because of the oxygen in the flame, as well as the lower particle velocity and longer spray distances which result in increased dwell time in the flame.

Some portion of the oxide is seen as smoke during the spray process, while the remaining portion is deposited in the coating. Samples of the smoke were collected and analyzed by X-ray diffraction. The main phases found were NiO, and gamma $Al_2O_3$. There was also a small amount of another compound possibly a spinel. This latter possibility was suggested by a slight shift of the gamma $Al_2O_3$ lines from their normal position. However, X-ray data on the sprayed coating shows no evidence of crystalline NiO, gamma $Al_2O_3$ or spinel. Apparently, the high quench rate of the depositing material has resulted in an amorphous or glassy structure. It is concluded that, although analysis of the smoke does indicate the formation of $Al_2O_3$ and NiO, the oxide as deposited is primarily non-crystalline. However, small percentages of crystalline oxides could go undetected as discussed for the non-stoichiometric NiAl.

BOND LAYER:

Interfacial studies in a plane cut at 4 deg to the hardened substrate show what appears to be an alloy layer distinct from either the steel substrate or the nickel aluminide coating. A typical layer can be seen in Fig. 11. This effect is similar to the findings of Allsop, et al.(8), with their work on sprayed molybdenum. Apparently, the substrate area immediately under a sprayed particle of the exothermically reacting nickel-aluminum is heated sufficiently to melt and fuse with the nickel aluminide, forming the alloy layer. The alloy zone varies in thickness but, in any case, is merely a thin surface effect. Further examination of the interface shows that the visible alloy bond is discontinuous, exists along about 50% of the interface line, and varies in thickness up to about 0.5u. Minimum detectable thickness by this technique was about 0.05u. The composition has not been determined, but it is probable that the layer is primarily a ternary alloy of Ni, Al and Fe.

In addition, results of the metallographic study parallel to the interface, illustrated in Fig. 12, also shown an apparent alloy layer confined to the area immediately beneath the center of an impacting particle. This effect was seen under 4 out of 23 particles observed through 20 polishing steps. This would be statistically consistent with an alloy layer 0.1u thick under all particles if each polishing step removed about 3u.

The question arose as to whether or not the layer visible in Fig. 12 is, in fact, some portion of the nickel aluminide particle which imbedded in the substrate. Reference is made to some work by the National Bureau of Standards where an analysis by Dr. Olive Engle(9) (concerning the damage caused by water droplets on leading edges of high-speed aircraft) was extended for application to flame spraying. Those workers used an equation for the lowest drop velocity at which damage to the metal will occur, i.e., the minimum velocity at which a liquid particle will imbed itself in a substrate upon impact.

An attempt was made to determine if nickel aluminide particles sprayed using the combustion process will, upon impact, imbed in a fully hardened SAE 1095 steel substrate. In this calculation the density of molten nickel aluminide was assumed to be 7.0 g/cm, and the velocity of sound through the liquid particle was taken to be about the velocity of sound through water (1.5 x $10^5$ cm/sec). Using these values in eq 1 (see Appendix), the lowest particle velocity at which a particle will imbed in a fully hardened steel would be about 10?,000 ft/sec. The average velocity of particles from the 5P flame spray gun is about 150 ft/sec. It appears that the order of magnitude of velocity necessary for damage is well beyond that which occurs in flame spraying, making it highly unlikely that a molten particle of nickel aluminide would penetrate the surface of the fully hardneed steel substrate, except by fusing with the surface layer.

The depth of the heat-affected zone in the substrate is clearly shown in Fig. 13. Associated with each impacting particle is a dark, crescent-shaped heat-affected zone, having its greatest depth of approximately 0.001 in. at the center and decreasing to the surface at the edge of the particle. The peak temperature decreases from the temperature of the molten alloy bond layer at the interface to some 400 to 500°F at the maximum depth of 0.001 in. where tempering of the martensite is visible. Figures 11, 12, and 13, are complementary and together show the full effect of a particle of nickel aluminide after it bonds to a steel substrate. Although the microstructures shown here were generated with 404 coatings, similar work was done with 450 and 405. The results were the same, therefore this discussion is equally applicable to the bonding characteristics of 450 and 405.

## CONCLUSIONS:

1. Combustion flame sprayed coatings of 404 contain approximately 60 to 70% $Ni_3Al$, or aluminum in solution in nickel and/or combinations of both, while plasma flame sprayed coatings contain 65 to 75%. Additionally, there is an estimated 10 to 20% nonstoichiometric nickel rich NiAl and/or a retained quenched form of nonstoichiometric NiAl in combustion and plasma flame sprayed 404 coatings.

2. Plasma sprayed and combustion sprayed coatings of METCO 450 are relatively soft. They contain approximately 90% nickel and nickel/aluminum solid solution. Only minor percentages of non-stoichiometric NiAl are found.

3. METCO 405 wire coatings are primarily non-stoichiometric NiAl, estimated between 80 and 90%. Some softer phases are found.

4. The remainder of all the coatings contain oxides of nickel and aluminum, these oxides being primarily amorphous.

5. Flame sprayed nickel aluminide coatings bond to most substrates by fusion, forming an alloy layer.

6. On steel the alloy bond layer is discontinuous, covering perhaps 50% of the surface, and varying in thickness up to 0.5u.

7. The heat-affected zone in ferrous substrates extends to a depth of approximately 0.001 in. immediately beneath the center of an impacting particle of nickel aluminide.

## ACKNOWLEDGEMENT:

The author wishes to thank Dr. H. S. Ingham, Jr. for his stimulating comments on the text of this paper and to Carlisle Small for metallographic preparation.

APPENDIX:

Specimen Preparation

1. Rough grind to a flat surface with a silicon carbide wheel.

2. Rough polish through 320, 400 and 600 grit silicon carbide at 1550 rpm, kerosene lubricant.

3. Polish with 9u diamond paste on Metcloth at 550 rpm, kerosene lubricant.

4. Polish with 1u diamond paste on metcloth at 550 rpm, kerosene lubricant.

5. For final polish a slurry of gamma alumina (0.1u) on Gamal cloth using the Vibromatic polisher.

Etchants: The nickel aluminide coatings were etched with either:

| Carapella's Reagent | Marbles's Reagent |
|---|---|
| 5 g ferric chloride | 4 g copper sulphate |
| 2 ml HCl | 20 ml HCl |
| 99 ml ethyl alcohol | 20 ml $H_2O$ |

X-Ray Diffraction Data

| Intensity | "d" Spacings |
|---|---|
| vw | 3.53 |
| m | 2.25 |
| vvs | 2.03 |
| w | 1.95 |
| ms | 1.76 |
| m | 1.375 |
| s | 1.245 |
| w | 1.175 |
| vw | 1.125 |
| vs | 1.065 |
| m | 1.018 |

where vw = very weak; w = weak; m = medium; ms = medium strong; s = strong; vs = very strong; vvs = very, very strong.

Equation 1

$$V_1 = \frac{19E^1(Z + Z^1)}{(pc^1Z^{13})1/2}$$

where $V_1$ = lowest drop velocity; $E^1$ = dynamic compressive yield strength of metal; Z = acoustic impedance of liquid particle (Z = cp) where c is velocity of sound through the liquid, p is its density; $Z^1$ = acoustic impedance of metal substrate; p = density of liquid particle; c' = velocity of sound through metal substrate.

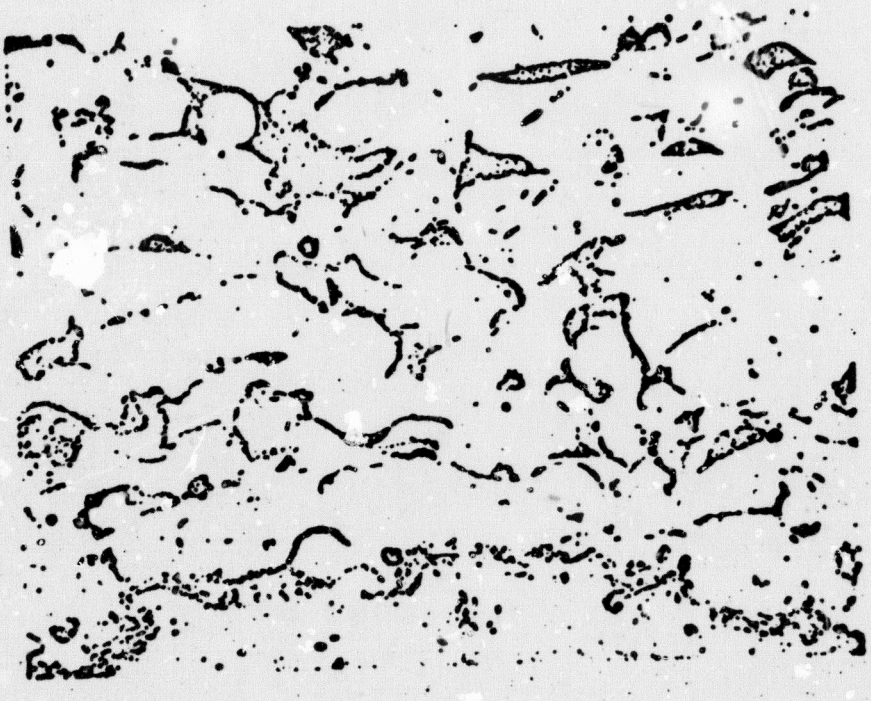

Fig. 1

A cross section through a ThermoSpray (SP) coating of METCO 404. Black areas are oxides. Hard NiAl particles may be identified by the slightly off white shading. Substrate is etched with Nital. Coating is unetched X300.

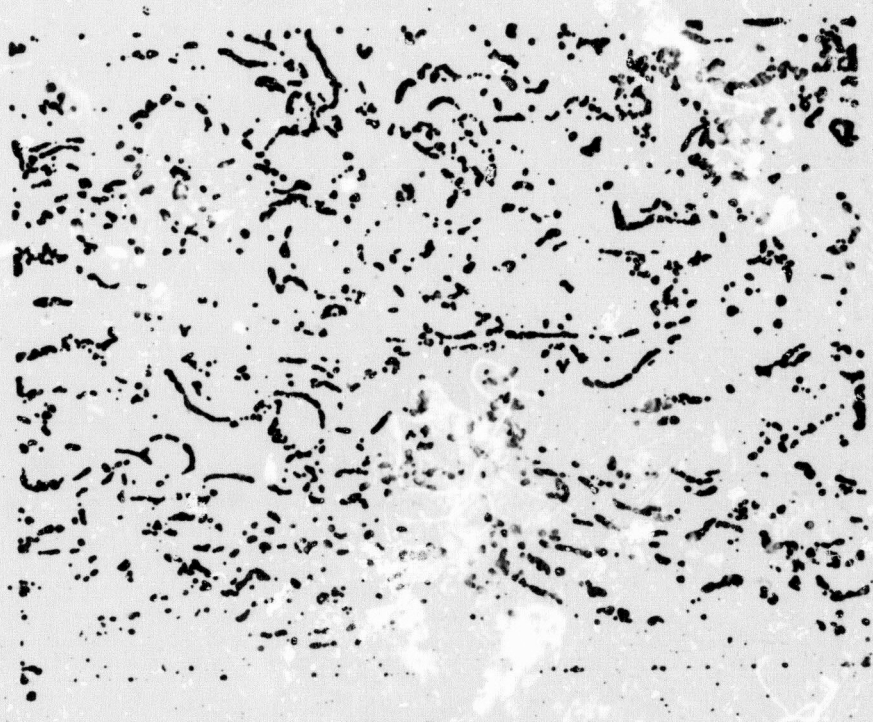

Fig. 2

Plasma sprayed 404 coating sprayed with nitrogen plasma. Black areas are oxides and off white particles are non-stoichiometric AlN. 300. Unetched.

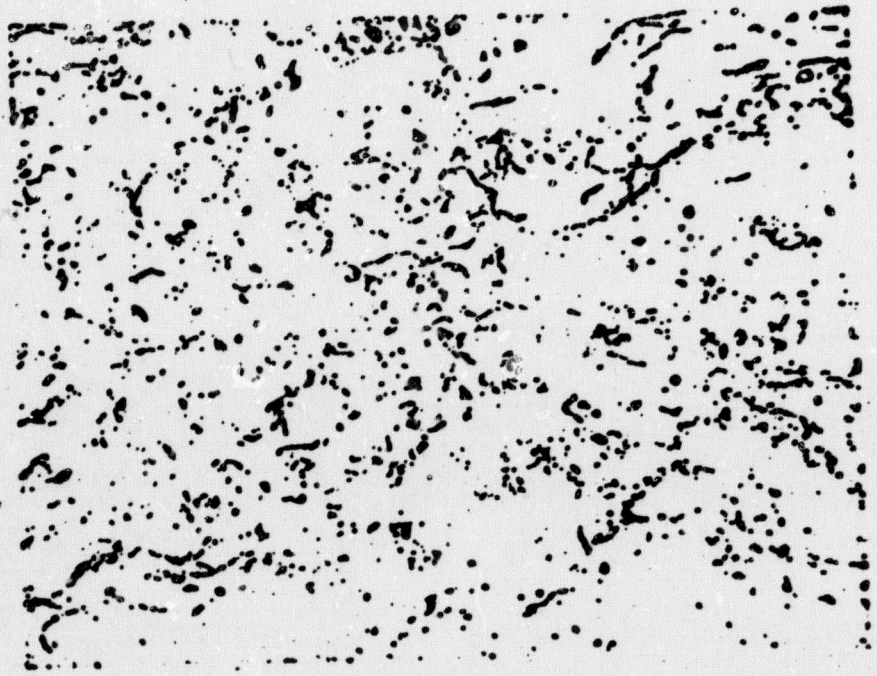

Fig. 3

Plasma 404 coating sprayed with argon plasma. Black areas are oxides and off white areas are non-stoichiometric NiAl. Unreacted aluminum can be identified as areas which are pure white with black specs. X300. Substrate etched with Nital.

Fig. 4

Photomicrograph of structure after melting and casting 10 g of METCO 404 powder. Shows the transformed non-stoichiometric NiAl (striated) and $Ni_3Al$ (unetched). Marbles reagent. X533.

Fig. 5

Example of transformed non-stoichiometric NiAl as found in 404 coating. The striated pattern is readily seen at this magnification; however, as may be noted in Fig. 1, the striations are not readily visible at lower magnification. Carapella's reagent. 1300X.

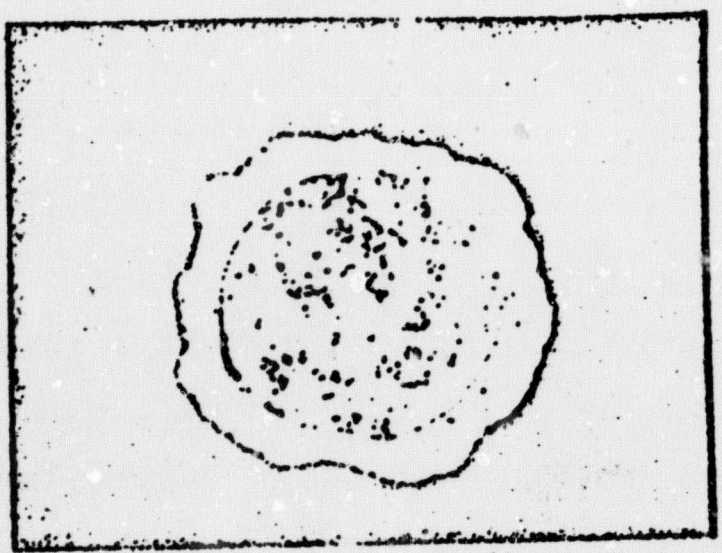

Fig. 6

Cross section through a #404 particle showing the aluminum core with continuous nickel shell.

TABLE 1 - Microhardness Data

| | Non-stoichiometric NiAl | | Al in solution in Ni or $Ni_3Al$ | |
|---|---|---|---|---|
| | $Khn_{50}$ | $R_A$[a] | $Khn_{50}$ | $R_A$[a] |
| Plasma flame sprayed 404 | 685 | 76 | 320 | 63 |
| Combustion flame sprayed 404 | 554 | 73 | 290 | 60 |
| | Composition 25 w/o Al As-cast | | Composition 13.3 w/o Al As-cast | |
| Hardness data by Grala E. M.[4] | $R_A$72 | | $R_A$53 | |

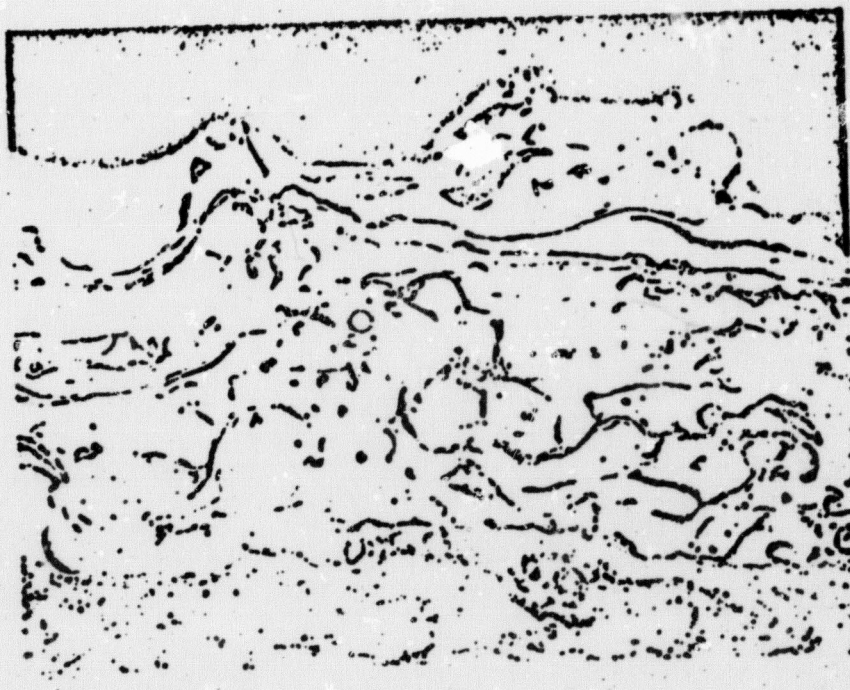

Fig. 7

Cross section through a 405 coating sprayed with an 8E gun. The non-stoichiometric NiAl particles can be identified by noting the large percentage of off white areas. X300. Substrate etched with Nital. Coating is unetched.

Fig. 8

Cross section through a ThermoSprayed (5P) coating of METCO 450. Black areas are oxides. Remaining major phase is nickel/aluminum solid solution. X300. Substrate etched with Nital. Coating unetched.

Fig. 9

Plasma coating of 450 sprayed with a Nitrogen plasma. Black areas are oxides. Off white areas identified as non-stoichiometric NiAl constitute less than 5% of the coating. The remaining particles are nickel and nickel/aluminum solid solution. X300. Substrate etched with Nital. Coating unetched.

E 981

Fig. 10

Plasma coating of 450, sprayed with argon plasma. This is almost the same as nitrogen sprayed except this coating contains fewer oxides; therefore the coating hardness is Rb 60.

Fig. 11

Example of the bond layer exposed on a polished plane cut at 4 deg to steel substrate. The bond layer is believed to be an alloy of Fe, Ni and Al. It is not continuous and is distinct from both the nickel aluminide particle (white) and the steel substrate (black). Steel etched with nital. X1333.

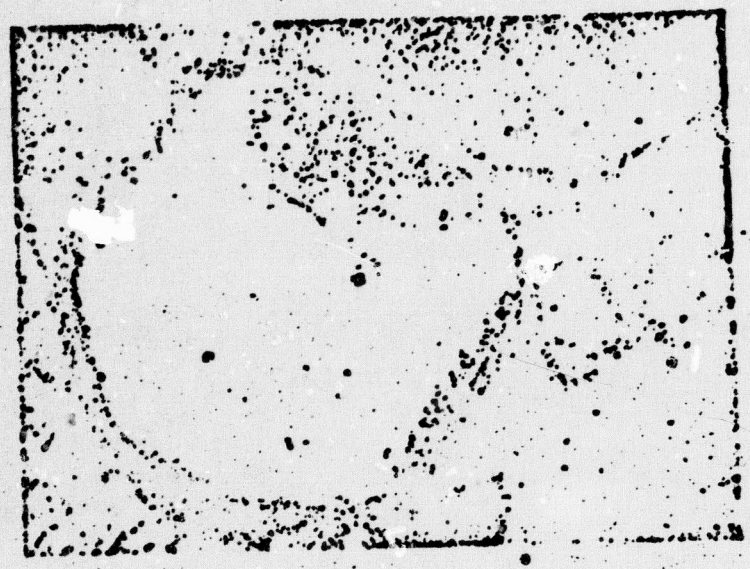

Fig. 12

A 90 deg cross section of a nickel aluminide coating on a polished surface of fully martensitic steel. The steel is etched with nital to reveal the depth of the heat-affected zone from one particle of nickel aluminide. The measured depth is approximately 0.001 in. at the center of the impacting particle and decreases toward the edge of the particle. X633.

Fig. 13

This photo shows a plane parallel to the substrate at or below the interface between the nickel aluminide coating and the martensitic steel substrate. Note that the entire area is martensitic except for a small area located at the center of the heat-affected zone. This white area is believed to be the alloy bond layer which formed by melting. X533.

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(1) Dittrich, F. J., Shepard, A. P., U. S. Patents 3,322,515 and 3,436,248.

(2) Dittrich, F. J., "New Flame Spray Technique for Forming Nickel Aluminide-Ceramic Systems", American Ceramic Society Bulletin, 492-496 (1965)

(3) Hansen, M., Constitution of Binary Alloys, McGraw-Hill Book Co., Inc. New York (1958).

(4) Guard, R. W., Turkalo, A. M., Mechanical Properties of Intermetallic Compounds, John Wiley & Sons, New York, J. H. Westbrook (Ed.), 141-159 (1960).

(5) Grala, E. M., Mechanical Properties of Intermetallic Compounds, John Wiley & Sons, New York, J. H. Westbrook (Ed.), 358-401 (1960).

(6) Bradley, A. J., Taylor, A., "An X-Ray Analysis of the Nickel-Aluminum System", Proc. Roy. Soc., London, A159, 56-72 (1937).

(7) Allsop, R. T., Pitt, T. J., Hardy, J. V., "The Adhesion of Sprayed Molybdenum", Metallurgia, 63, 125-131 (1961).

(8) Moore, D. G., Eubanks, A. G., Thornton, H. R., Hayes Jr., W. O., Crigler, A. W., "Studies of the Particle-Impact Process for Applying Ceramic and Cermet Coatings", National Bureau of Standards, Appendix I (August 1961).

(9) Engle, Olive G., "Pits in Metals Caused by Collision with Liquid Drops and Soft Metal Spheres", National Bureau of Standards, 1959, J. Res. 62, 6, 229.

E 986 PX-50

July 1, 1960

Mr. R. F. Pearce, Project Engineer
Head, Industrial Dept.
Sherritt Gordon Mines Limited
Fort Saskatchewan, Alberta

Dear Mr. Pearce:

We are sending today, to your attention, approximately 10-1/2 pounds of special aluminum powder via Railway Express - Receipt No. 57-89-43. An open purchase order is enclosed to cover the charges.

The atomized aluminum powder is separated into five particle size ranges, as follows:

| | | |
|---|---|---|
| (1) | -140 +170 | 2.0 oz. |
| (2) | -170 +200 | 29.0 oz. |
| (3) | -200 +230 | 32.5 oz. |
| (4) | -230 +270 | 64.5 oz. |
| (5) | -270 +325 | 40.0 oz. |

We would like to have this powder coated with nickel, to give a finished powder which is the following composition by weight:

| | | |
|---|---|---|
| Aluminum | 17.5% | +1/2% -1 1/2% |
| Nickel | Remainder | |

We believe that this composition is the optimum for our purpose, and would like your advice as to whether or not you can hold the above tolerance on the aluminum content of the finished powder. We would also like to have the powder demagnetized after final processing.

At your convenience, will you let me know when I may expect to receive the finished powder?

Yours very truly,

METALLIZING ENGINEERING CO., INC.

F. J. Ditrich
Project Engineer, R & D

FJDip

00177

E 987

K385
T488

PX-51

# SAM TOUR & CO., Inc.

ENGINEERS · METALLURGISTS · CONSULTANTS

44 TRINITY PLACE

NEW YORK 6, N. Y.

WHITEHALL 3-3158

April 13,1949

Mr. H. S. Ingham,
Metallizing Engineering Co., Inc.,
38-14 -- 30th St.,
Long Island City, N. Y.

Dear Herb:

Enclosed are two photostat copies of a data sheet on the Aluminum-Nickel System.

You will note that 31.5% Al gives the 3000° F. compound with nickel.

Very truly yours,

Sam Tour

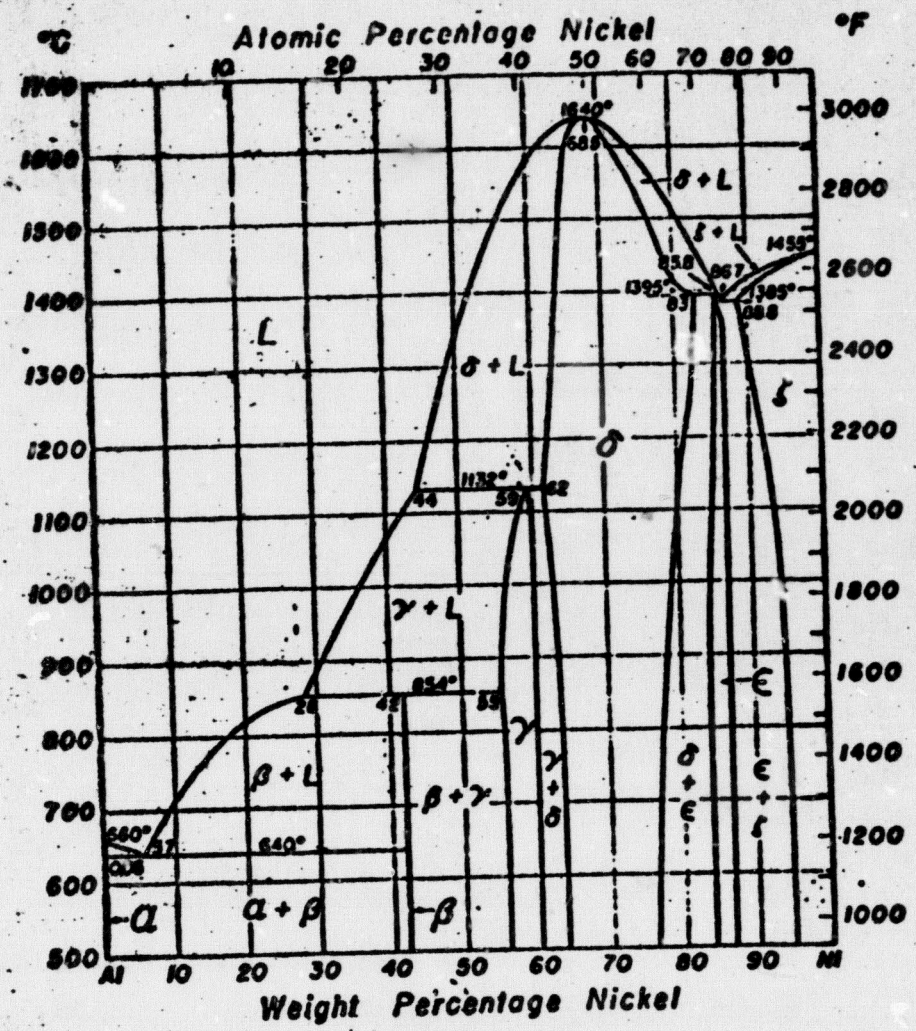
Sam Tour

ST:AR
Enc.

# E 988

## Al-Ni Aluminum-Nickel

By W. L. Fink[1] and L. A. Willey[2]




The diagram shown is from investigations by Alexander and Vaughan,[1] Schramm,[3] and Fink and Willey.[4]

The Curie temperature of nickel is decreased linearly from 360 to 70 C by the addition of 4.8% Al in solid solution.[3]

The solubility of nickel in solid aluminum is, as follows: 0.040% at 625 C, 0.016 at 600, 0.006 at 585. The solubility of aluminum in solid nickel is 9.7% at 1300 C, 8.0 at 1200, 6.4 at 1000, 6.0 at 900, 5.0 at 700

Nickel is used as an alloying element in a few commercial aluminum-base alloys principally in aluminum-copper-magnesium alloys and aluminum-silicon-magnesium alloys. Nickel increases the strength of these alloys at elevated temperature and decreases the coefficient of thermal expansion, but usually decreases the resistance to corrosion.

At the other end of the system there are a few commercial alloys that are used principally for thermocouple wires.

### References

1 W. L. Fink and L. A. Willey, Trans. AIME, 133: 248 (1939).

2 W. O. Alexander and C. J. Vaughan, J. Inst. Metals, 66: 161 (1940).

3 J. Schramm, Z. Metallkunde, 31: 241 (1939).

4 A. J. Bradley and A. Taylor, Proc. Roy. Soc. (London), A159: 56 (1937).

* Aluminum Research Laboratories, New Kensington, Pa.

**Crystal Structures and Lattice Parameters of Aluminum-Nickel Alloys**

| Phase | Crystal Structure | Lattice Parameter, kX units: a | b | c |
|---|---|---|---|---|
| θ | Orthorhombic | 6.560 | 7.332 | 4.372 |
| β | Trigonal | 4.028 | 4.384 | ... |
| γ | Body-centered cubic | 2.864 | .... | ... |
| δ | ... | 2.88 | .... | ... |
| α | Face-centered cubic | | | |

An intermetallic Ni-Al compound is present at 1640°C. The formula ascribed to this compound is NiAl, i.e. 50% atomic percentage of Ni and Al each and 68.5% Ni and 31.5% Al by weight.

00593

E 989

00625

## Metal-coated metal and ceramic powders . . .

. . . that promise improved properties for powdered-metal and metal-ceramic parts are soon to be made available on a semicommercial basis from Sherritt Gordon Mines Ltd, Fort Saskatchewan, Alberta, Canada.

These are metal and nonmetal powders coated with nickel, cobalt, copper and other metals by gaseous hydrogen precipitation from an ammoniacal solution at temperatures of 300 to 400 F and pressures of 200 to 500 psi. Particles ranging all the way from less than one micron to several hundred microns can be coated this way, and lend themselves to roll-compacting (direct production of metal strip from powder) as well as to the standard pressing and sintering techniques.

A special advantage of the new coated powders, says V. N. Mackiw, S-G's R & D director: They make it possible to introduce small percentages of very fine, hard, nonmetal phases uniformly throughout the matrix metal, thus providing high-quality dispersion-hardened alloys. One example is a nickel-3% chrome oxide material which S-G has successfully roll-compacted, using techniques previously developed for and applied to production of pure nickel strip

PX-53

E 990

00626

F. J. D.

February 24, 1959

Sherritt Gordon Mines, Ltd.
Fort Saskatchewan
Alberta, Canada

Attention: Mr. V. N. Muckiw, R & D Director

Dear Sirs:

We read with much interest in the R & D Edition of PRODUCT ENGINEERING the announcement that metal coated powders would soon be made available.

We are manufacturers of powder spray and wire metallizing equipment and are very much interested in any information and trial samples of metal coated ceramic powders we can secure. We can supply the ceramic powders in the proper mesh size for use in our equipment.

Can alumina ($Al_2O_3$) and stabilized zirconia ($ZrO_2$) powders in the general particle size range of 60u to 10u be coated with, for instance, nickel or cobalt? What is the approximate thickness particles in this size range could be coated. Would the resultant material be composed of individual metal coated ceramic particles that are free flowing powder, or would there be a metal-ceramic compact that would have to be milled to powder? What would be approximate cost of coating a pound of ceramic power to a thickness of approximately .001" with nickel or cobalt be? Could chromium boride powder or titanium boride powder be coated with, for instance, nickel?

Any information you can give us will be greatly appreciated. It is entirely possible that, with little development work, composite powders for coatings could be developed which would be to our mutual benefit.

Yours very truly,

METALLIZING ENGINEERING CO., INC.

F. J. Dittrich
Project Engineer

FJD:p

E 991

PX-60

Internal Correspondence
Finishing

Eutectic + Castolin Institute
for the Advancement of Maintenance
and Repair Welding Techniques®
PERMANENT TECHNICAL ASSISTANCE
Exclusive Processes • Wearfacing Alloys • EutecRod® Alloys • EutecTrode® Alloys
Continuous Electrodes • Powdered Alloys • Chemical Aids and Fluxes

Date April 20, 1972
To Mr. M. Patel
From Mr. B. Brachfeld
Copies Mr. J. F. Quaas

Subject
RESULTS OF X-RAY DIFFRACTION STUDY OF Ni-Al POWDER SPRAYED INTO AIR
EUTECTIC 29904 & METCO 450

A previous X-ray diffraction study of Ni-Al powder sprayed on test slugs found no Ni-Al compound to be present. In this study, instead of being sprayed onto test slugs, the powder was sprayed into the air and collected in a bucket. In this way the powder would be cooled down to room temperature at a slower rate than previously, which might enhance the formation of a Ni-Al compound.

X-ray diffraction analyses of Eutectic 29904 and Metco 450 powders, prepared in the above manner, revealed the presence of Ni, Al and their respective oxides. Once again, no Ni-Al compound could be found.

BB:hpc

50429

PX-62

E 992

January 20, 1972

Mr. J. F. Quass
Mr. M. Patel
Mr. H. Kapralos

COLORING EXOTEC 29904

The following three RoToTec sprayed coatings were sent to E. F. Fullam, Inc. for microprobe analysis:

| Identification | Description Of The Coating |
|---|---|
| Sample 1 | Standard Exotec 29904 |
| Sample 2 | 2% $PbCrO_4$ coated on standard Exotec 29904 |
| Sample 3 | 1% $PbCrO_4$ coated on standard Exotec 29904 |

The xerox copy on the report from E. F. Fullam on the results is attached.

Findings:

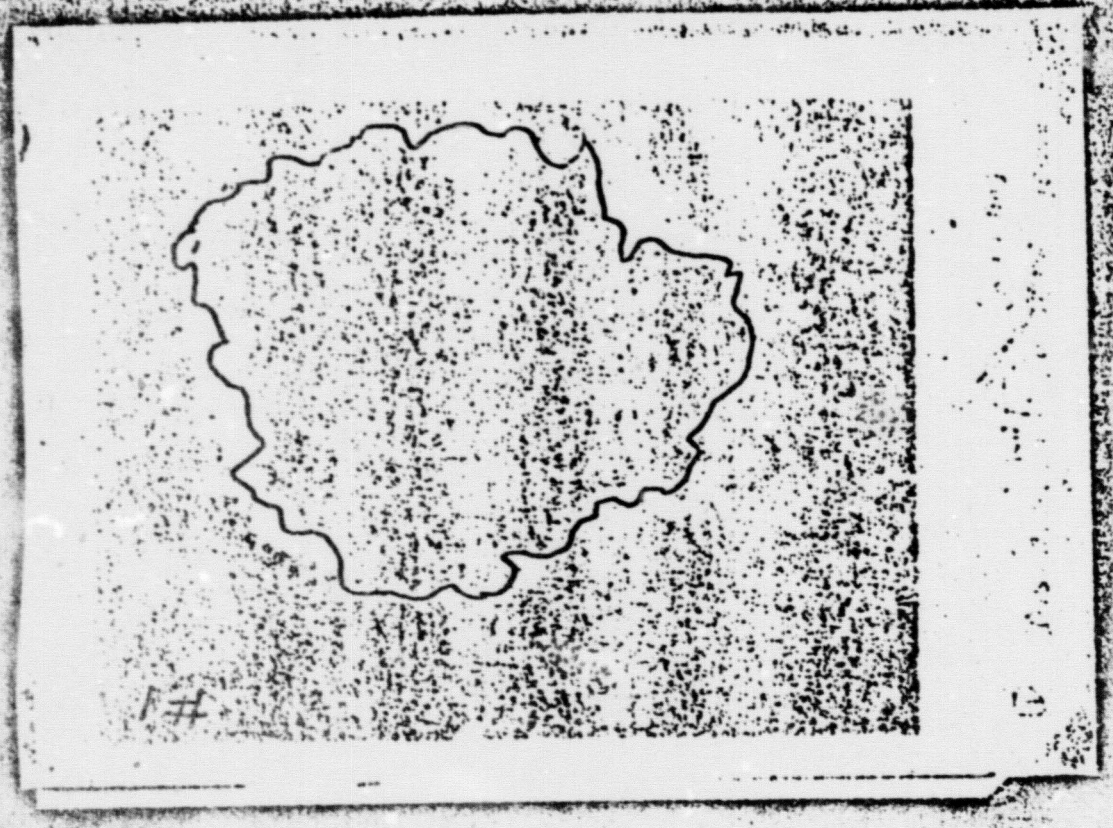
1. The standard Exotec sprayed coating consists mainly of elemental nickel with a trace amount of $Al_2O_3$.
2. The addition of $PbCrO_4$ in the range of 2-3% does not change overall chemistry of Exotec 29904 sprayed coating.

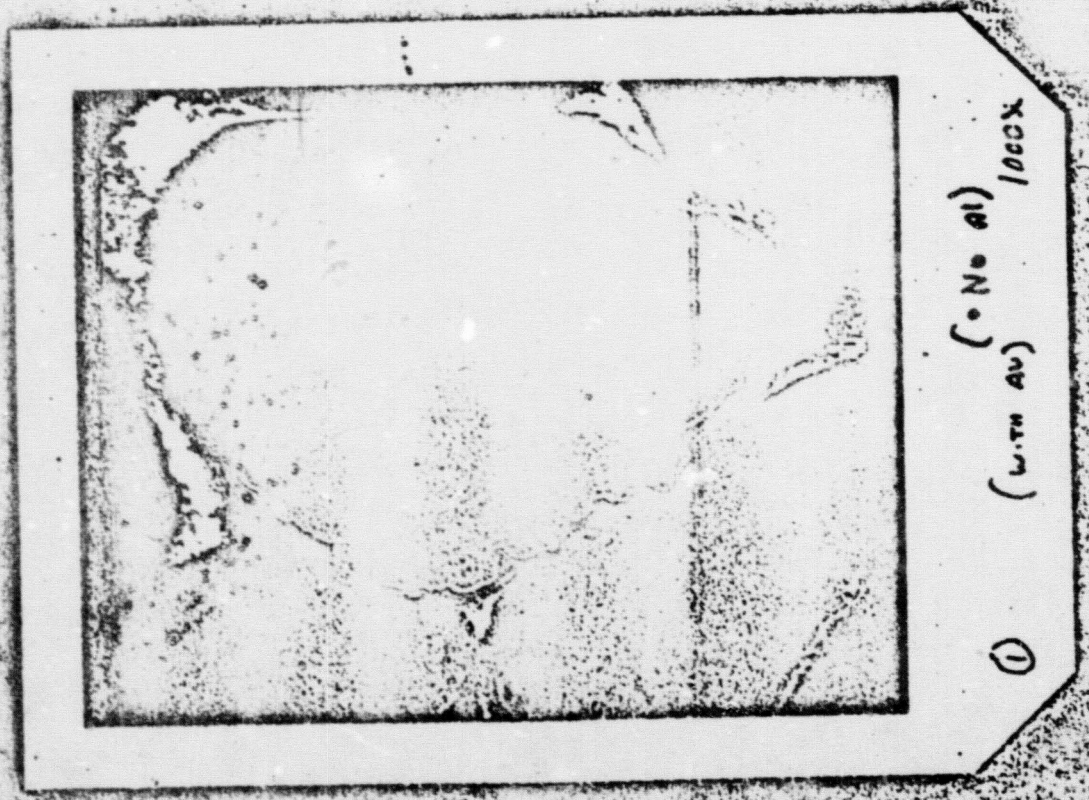
If you require any further information, please advise.

HP:hpe
att.

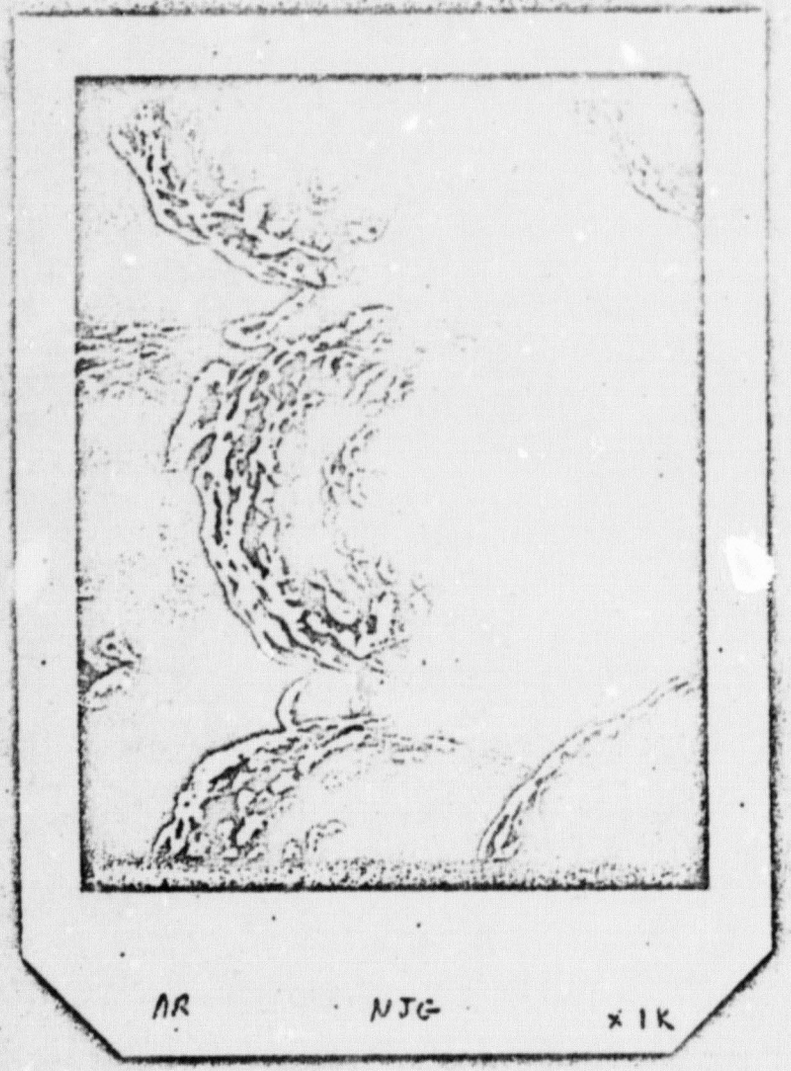
E 993

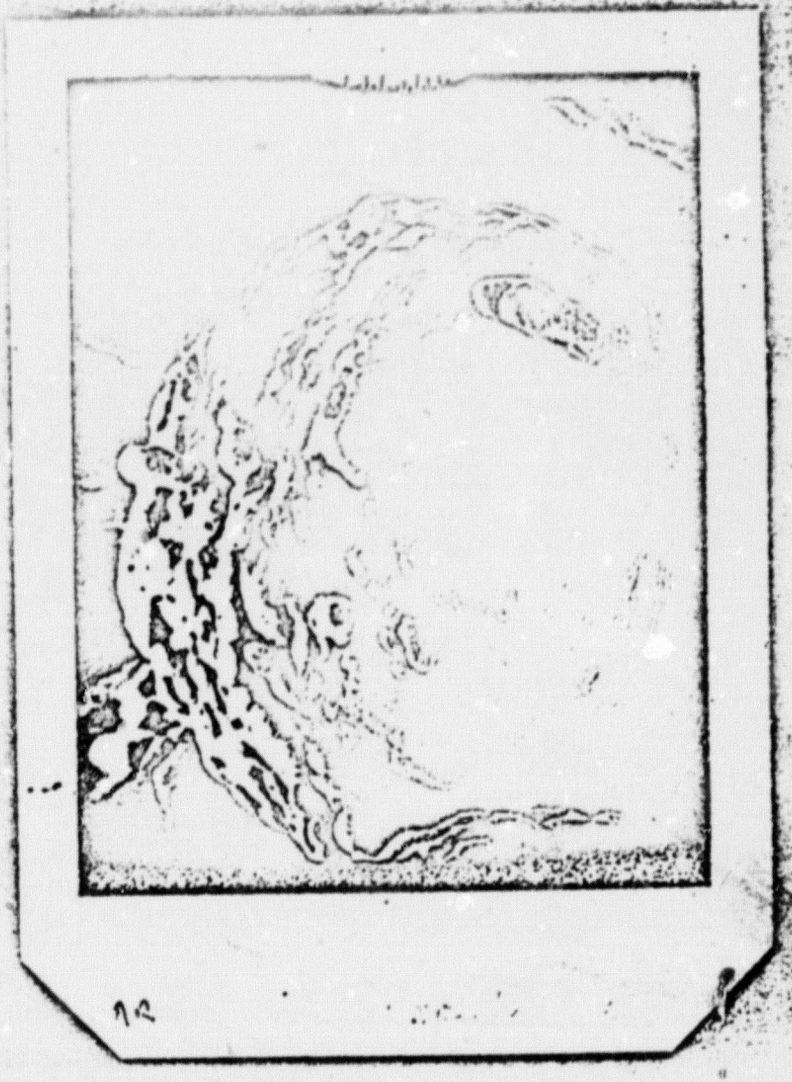
PX-63





(1) (With Al) (No Al) 100X




E 994

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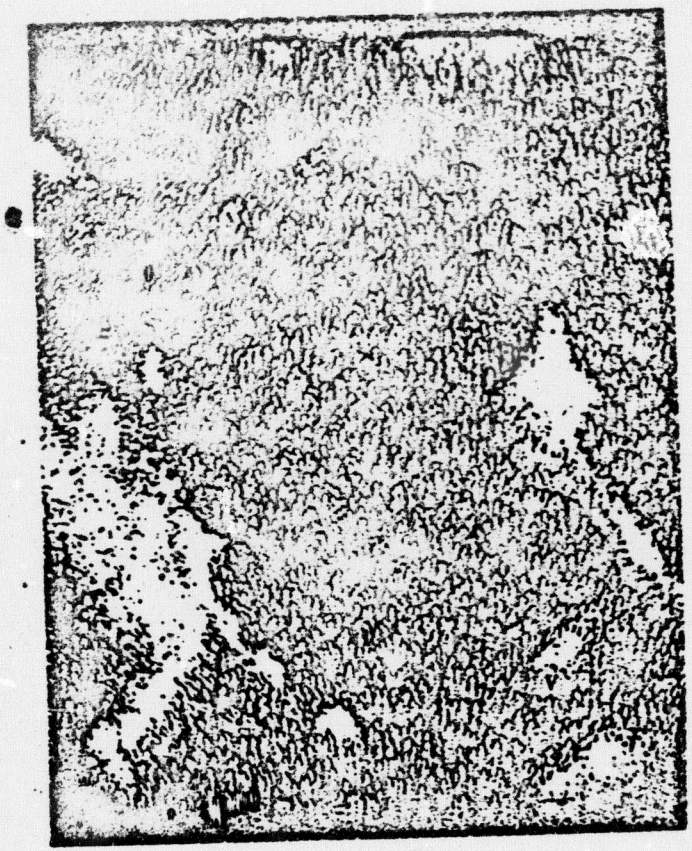
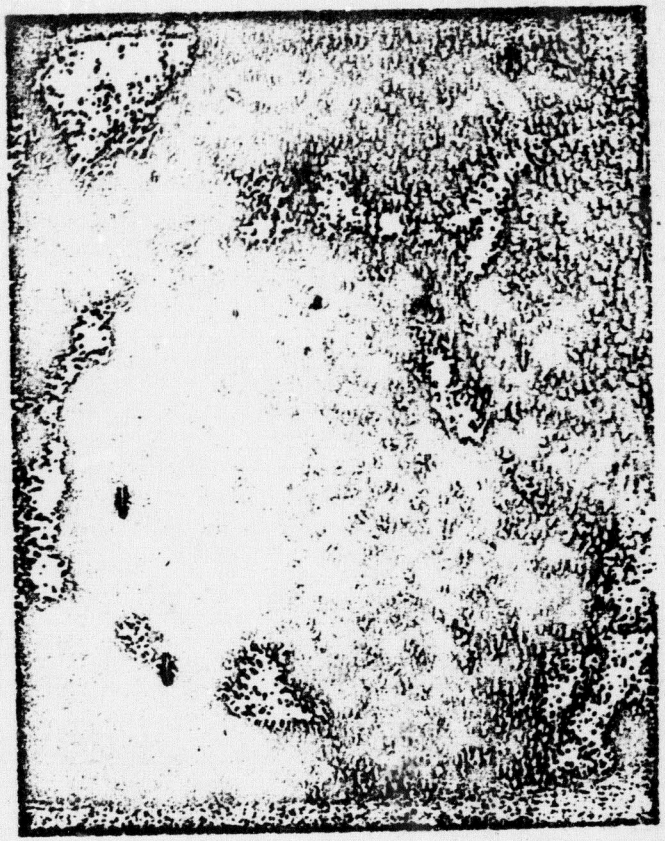


(2) 10000X

E-985

(3) WITHOUT Al. (No Al.) 1000X



3 (WITHOUT Au) 1000X

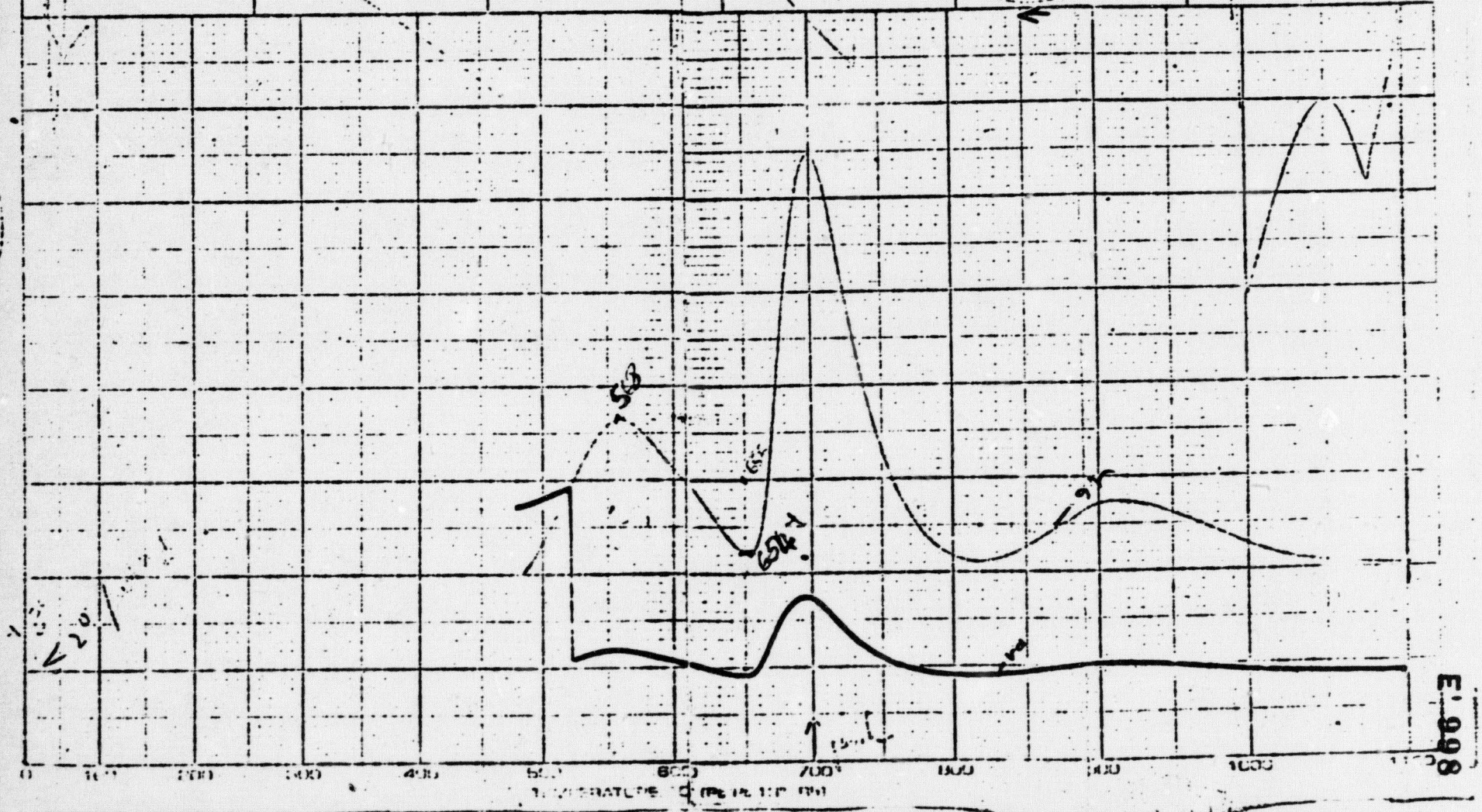
E 996

E 997

PART NO. 990972

72-64

| | | | | | |
|---|---|---|---|---|---|
| RUN NO. 2 DATE 10/15<br>OPERATOR Bronwyn<br>SAMPLE Eastaki 2901 9515<br>ATM Air<br>FLOW RATE | T-AXIS<br>SCALE, mv/in 0.5<br>PROG RATE, °C/min 50<br>HEAT X COOL ISO<br>START, in 0 | DTA-DSC<br>SCALE, °C in 10<br>(mcal/sec) /in 2<br>WEIGHT, mg 72<br>REFERENCE $Al_2O_3$ | TGA<br>SCALE, mg/in<br>SUPPRESSION, mg<br>WEIGHT, mg<br>TIME CONST., sec<br>dY, (mg/min) /in | TMA<br>SCALE, mils/in<br>MODE<br>SAMPLE SIZE<br>LOAD, g<br>dY, (10X), (mils/min) /in | 83 S.C@50 = 50 / RA=0<br>570°C → 1001°C |




E-998

E 999

$\frac{30}{1.7} \approx 11^{\circ}/\text{mm}$

$\frac{3.2}{1.7}$

E 998

E1000

$\frac{9°}{.5} = 10°/m$

PART NO. 970072

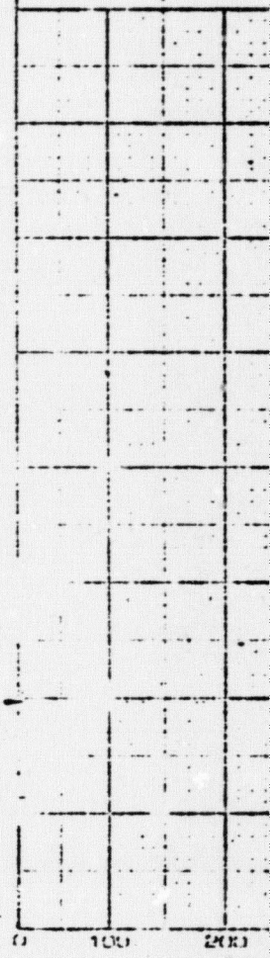
RUN NO 8 DATE 10/17

OPERATOR Vitek

SAMPLE Metro 80/20

ATM Air @ 1atm

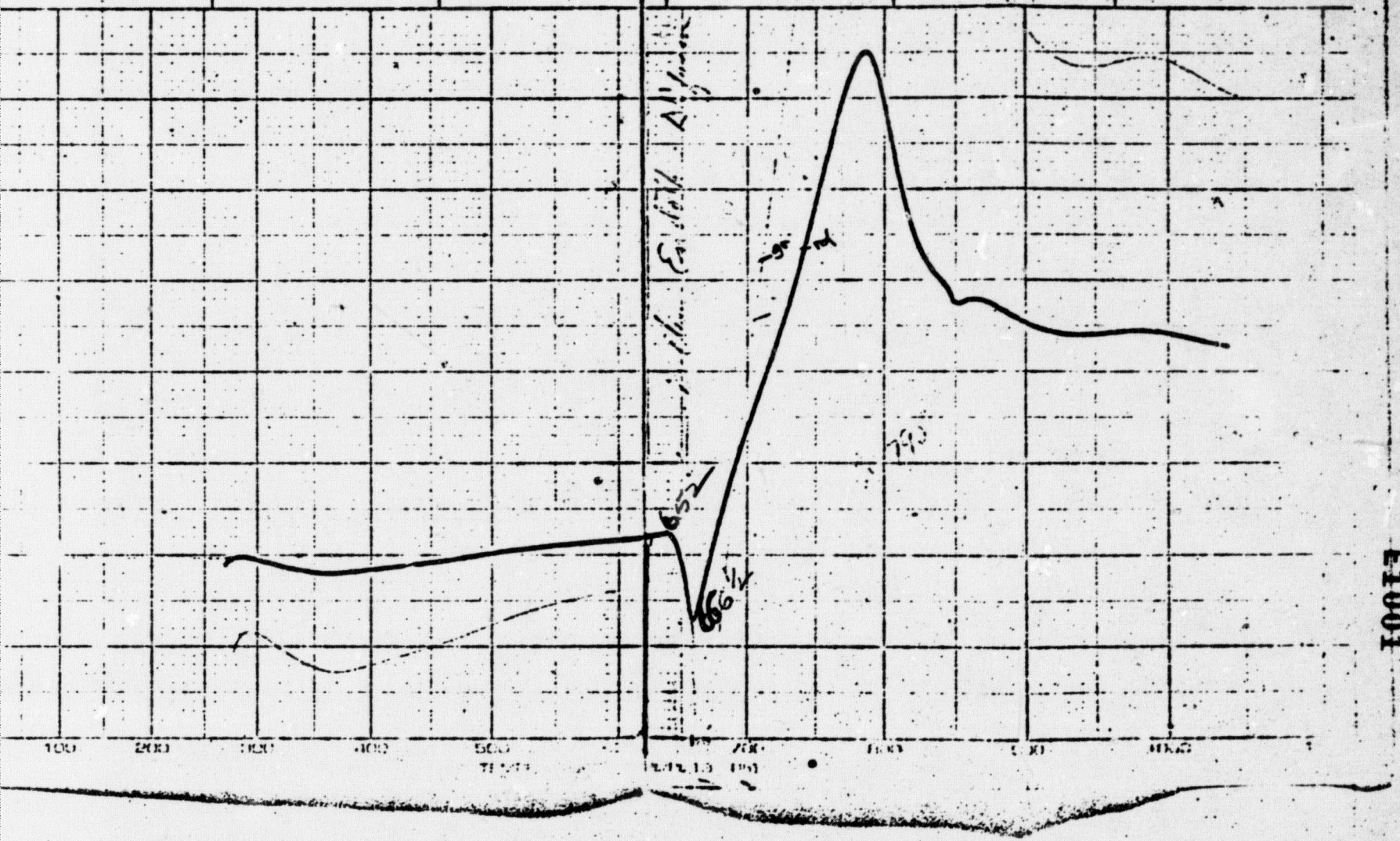
FLOW RATE —



ART NO. 990072

PE-65

| | T-AXIS | DTA-DSC | TGA | TMA | |
|---|---|---|---|---|---|
| RUN NO 8 DATE 10/17 | SCALE, mv/in 0.8 | SCALE, °C/in 25 | SCALE, mg/in | SCALE, mils/in | Scan no 6 |
| OPERATOR Vikk | PROG. RATE, °C/min 50 | (mcal/sec)/in | SUPPRESSION, mg | MODE | |
| SAMPLE Metro 80/20 | HEAT X COOL 80 | WEIGHT, mg 72 | WEIGHT, mg | SAMPLE SIZE | |
| ATM Air + 1 atm | SHIFT, in 0 | REFERENCE $Al_2O_3$ | TIME CONST., sec | LOAD, g | |
| FLOW RATE — | | | dY, (mg/min)/in | dY, (10X), (mils/min)/in | |




E1001

E1002

PX-66

# CONSTITUTION OF BINARY ALLOYS

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Second Edition
*Prepared with the Cooperation of*
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Metals Research Department
Armour Research Foundation, Chicago, Illinois

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1958

3. W. L. Fink, L. A. Willey, and H. C. Stumpf, *Trans. AIME*, 175, 1948, 361-371.
4. E. Scheuer, *Z. Metallkunde*, 27, 1935, 83-85. Al of 99.7% purity was used.
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I.4630
0.4370

## Al-Nb Aluminum-Niobium

The crystal structure of the compound $NbAl_3$ (53.44 wt. % Nb) [1] is tetragonal of the $TiAl_3$ ($D0_{22}$) type, with $a = 5.438$, $c = 8.601$ A, $c/a = 1.582$, 4 formula weights per unit cell [2]. X-ray investigation of a Nb-rich alloy with approximately 10 at. (~3 wt.) % Al [3] did not reveal the composition of the intermediate phase coexisting with Nb or a Nb-rich solid solution [4].

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I.3710
0.7281

## Al-Nd Aluminum-Neodymium

The compound NdAl (84.25 wt. % Nd) has the structure of the CsCl (B2) type, $a = 3.74$ A [1].

1. C. W. Stillwell and E. E. Jukkola, *J. Am. Chem. Soc.*, 56, 1934, 56-57.

I.6628
0.3378

## Al-Ni Aluminum-Nickel

Since the first investigation of the phase diagram [1], which established its principal features, phase relationships have undergone considerable changes in parts, especially above 50 at. % Ni. The diagram presented in Fig. 68 is largely based on the work by [2] [0-18 wt. (0.2 at.) % Ni], [3] (X-ray analysis of the whole system using slowly cooled powders), [4] (thermal and micrographic analysis of alloys with 5-95 wt. % Ni), [5] (thermal and micrographic analysis of alloys with more than 75 wt. % Ni), [6] (formation of the $Ni_2Al$ phase), and [7] (phase boundaries in the range 70-90 at. % Ni).

Alloys with 0-50 At. % Ni. The partial phase diagram is well established. There is good agreement as to the liquidus curve [2, 4, 8]. The eutectic was placed at 6 wt. (2.85 at.) % Ni, 630°C [1]; 5.3 wt. (2.5 at.) % Ni, 633°C [9]; 5.7 wt. (2.7 at.) % Ni, 640°C [2]; and 6.3-6.4 wt. (3.0-3.06 at.) % Ni, 640°C [10]. The solid solubility of Ni in Al was found to be about 0.05 wt. (0.023 at.) % at 640°C, 0.028 wt. (0.013 at.) % at 600°C, and 0.006 wt. (0.003 at.) % Ni at 500°C [2]. In good agreement, [11] found 0.01-0.02 wt. (0.005-0.009 at.) % Ni at 560°C.

$NiAl_3$ (42.03 wt. % Ni) [12, 2, 3, 13, 4] is of singular composition [3, 13]. Its temperature of peritectic formation was determined as 812°C [1] and 854°C [9], with the peritectic melt containing about 28 wt. (15.1 at.) % [1] or 28.4 wt. (15.3 at.) % Ni [4].

The intermediate phase formed peritectically at 1132-1133°C [1, 4] was first suggested to be $NiAl_2$ [1] but later identified as based on the ideal composition $Ni_2Al_3$ (59.19 wt. % Ni) [3, 13, 4]. Its range of homogeneity [55.3-60 wt. (36.3-42.8 at.) % Ni] was determined micrographically at temperatures between 1120 and 600°C [4]; see also [3]. The $\beta'$ phase, based on the composition NiAl (68.51 wt. % Ni), has a

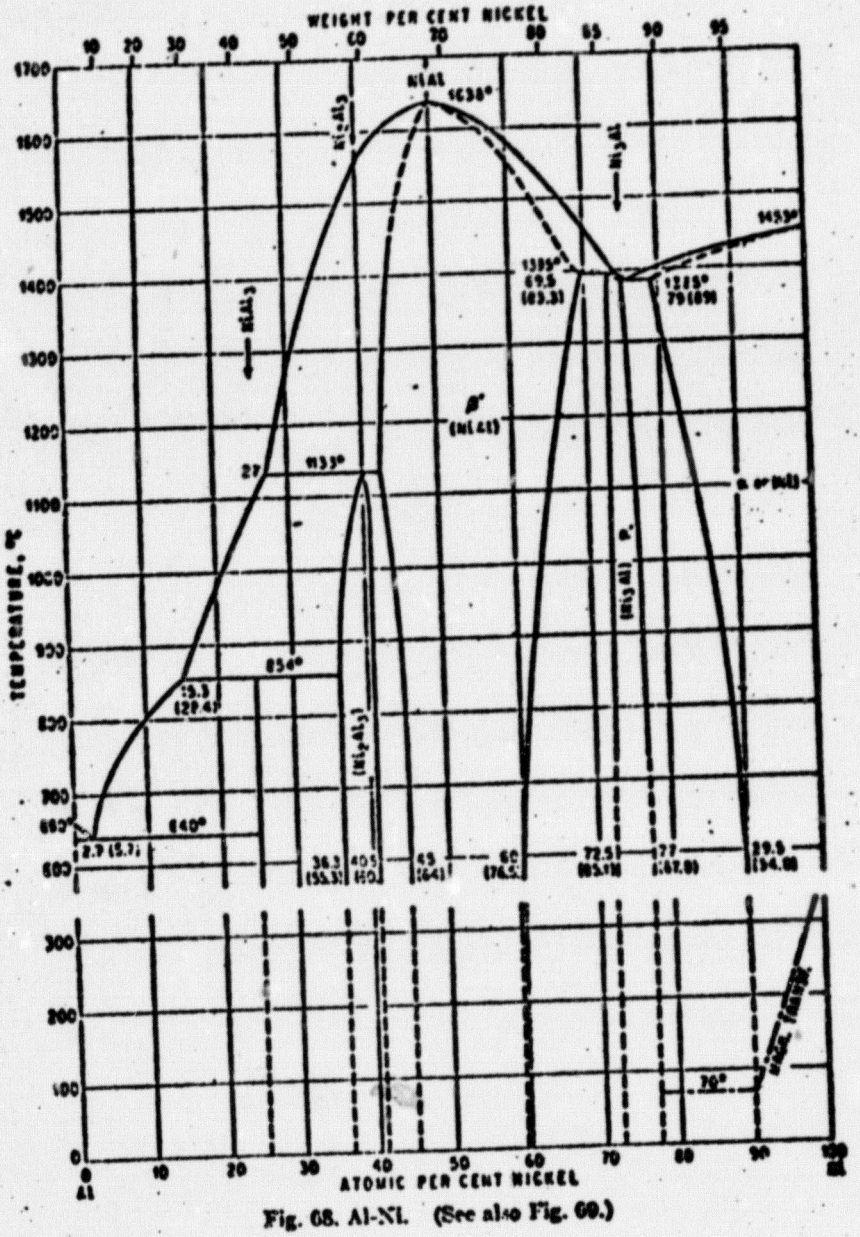


Fig. 68. Al-Ni. (See also Fig. 69.)

maximum melting point of 1638°C [4]. Its boundary on the Al-rich side was determined micrographically for temperatures between 1420 and 600°C [1]; see also [3].

Alloys with 50-100 At. % Ni. The constitution in this range of composition, which proved to be much more complicated than was assumed earlier [1], was the subject of numerous investigations [14, 15, 3, 4, 16, 5, 17, 6, 7]. The existence of the phase based on the ideal composition $Ni_2Al$ (86.71 wt. % Ni), first detected by [14], was confirmed by all later investigators, with the exception of [16]. There is great discrepancy, however, as to the equilibria involving its formation, its range of homogeneity, as well as to the $\beta'/(\beta' + \alpha')$ and $\alpha/(\alpha + \alpha')$ boundaries.

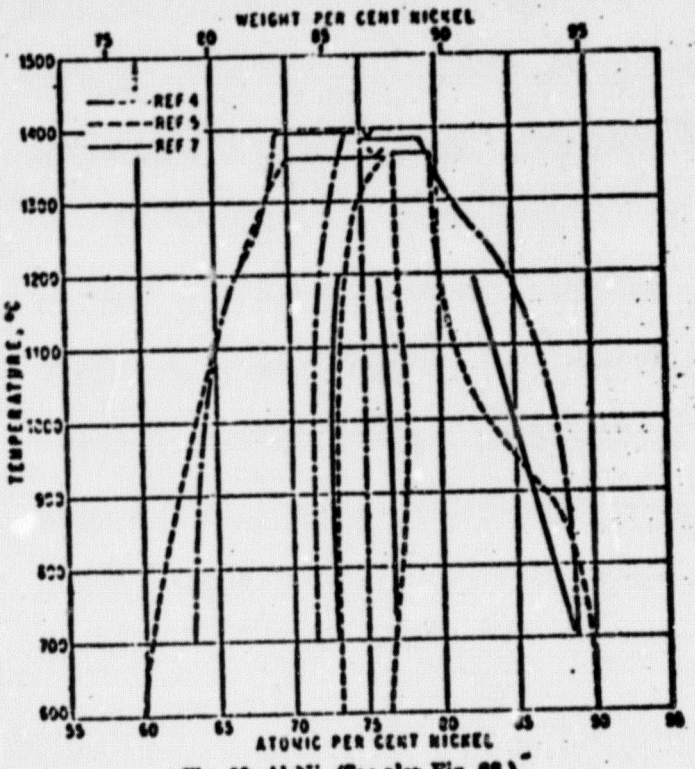


Fig. 69. Al-Ni. (See also Fig. 68.)

The diagrams given by [14] and [16] are highly improbable and incompatible with all later findings. [14] found phases of compositions $Ni_3Al$ and $Ni_4Al$; and [16] concluded that the $Ni_4Al$ phase was, in fact, $Ni_3Al$ and that the $Ni_4Al$ phase did not exist.

[3] suggested that the $Ni_3Al$ ($\alpha'$) phase is continuous with the $\alpha$ phase at temperatures above 1105°C, i.e., that the $\alpha + \alpha'$ field takes the form of a closed loop. This was not confirmed, however, by [3-7, 17]. Whereas the diagram by [4] shows the two three-phase equilibria liq. + $\beta' \rightleftarrows \alpha'$ at 1395°C and liq. $\rightleftarrows \alpha + \alpha'$ at 1383°C, [5] concluded that the reactions occurring at the solidus were liq. + $\alpha \rightleftarrows \alpha'$ at 1382°C and liq. $\rightleftarrows \alpha' + \beta'$ at 1360°C. This discrepancy appears to have been cleared up [6] in favor of the results by [4].

The phase boundaries in this region are presented in Figs. 68 and 69. In the main diagram (Fig. 68) the three-phase equilibria by [4] and the phase boundaries by [5, 7] have been adopted, with the exception of the $\alpha/(\alpha + \alpha')$ boundary, which represents a compromise. The phase boundaries suggested by [17] were not considered, as they were obtained by extrapolation from an attempt to smooth out the boundaries within the ternary system Al-Fe-Ni.

Temperatures of the magnetic transformation of the $\alpha$ phase were determined by [1, 18, 19, 5]; those by [5] were adopted in Fig. 68.

Crystal Structures. $NiAl_3$ has an orthorhombic structure and is the prototype of the $D0_{11}$ type, $a = 6.611$ A, $b = 7.366$ A, $c = 4.812$ A [13, 3]; see also [20, 21]. $Ni_2Al_3$ is hexagonal, of the $D5_{13}$ type, $a = 4.036$ A, $c = 4.900$ A, $c/a = 1.214$, at the stoichiometric composition; for change of parameters with composition see [13, 3]. NiAl ($\beta'$) is b.c.c. of the CsCl ($B2$) type, $a = 2.857$ A at 49.8 at. % Ni [3]; for change of parameter with composition see [3, 22]. See also earlier work by [23, 15, 24] and further studies by [25-27]. The $\alpha'$ phase ($Ni_3Al$) is f.c.c. of the $Cu_3Au$ ($L1_2$) type [15, 3, 4, 21], $a = 3.589$ A at 75 at. % Ni [3]. Lattice parameters of the $\alpha$ phase have been reported by [15, 3].

Supplement. [28] measured the electrical resistivity and thermal dilatation of alloys containing up to 35 wt. % Al. The isotherms of these properties exhibit sharp minima at the compositions NiAl and $Ni_3Al$. From the dilatation data, [28] suggest that $Ni_3Al$ undergoes a solid-state transformation at about 600°C.

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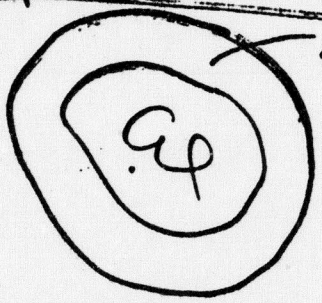
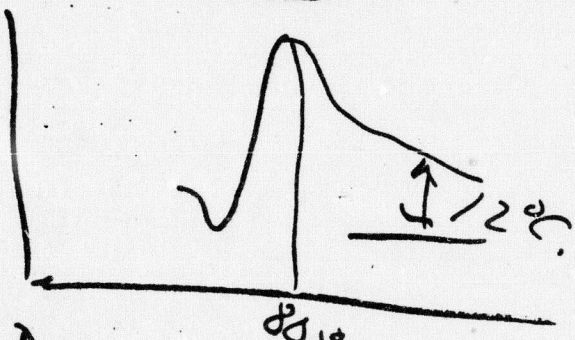
# 404

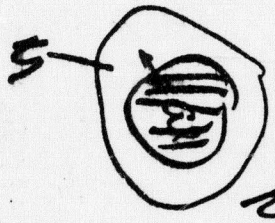
80 Ni - 20 Al

PATENTED



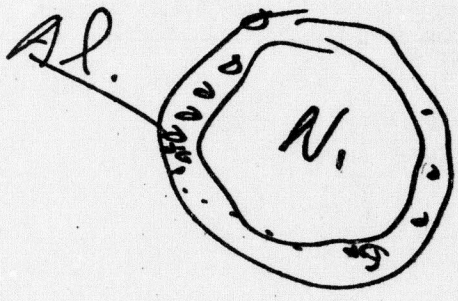

ΔH Evolved Liquid forming

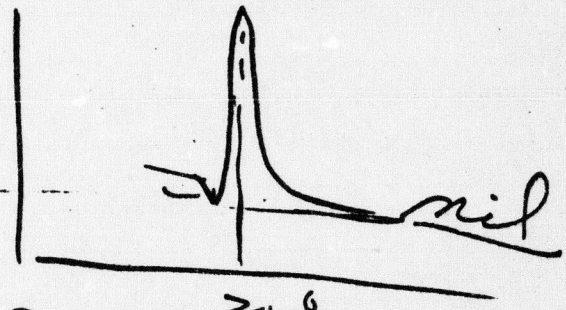



# 29904 & Metco 450

Ni 95 - 5 Al

ACCUSED




Exothermic - oxidation

Al + O = Al2O3

660°c

Al + O = Al2O3

ΔH -

PX-69

PLAINTIFF'S EXHIBIT
IDN 70
22 5.102



| Group → | IIIa | IVa | Va | VIa |
|---|---|---|---|---|
| Series ↓ | | | | |
| 2 | B ✓ | C | N | O |
| 3 | Al ✓ | Si ✓ | P | S |
| 4 | Ga ✓ | Ge ✓ | As ✓ | Se ✓ |
| 5 | In ✓ | Sn ✓ | Sb ✓ | Te ✓ |

Rods are listed as example by Dittrich

Hume Rothery states

"In Intermetallic cpds most strongly electronegative element plus strongly electropositive elements (Those are Ni, Co, Fe, Cu, etc)

∴ more stable I-M cpds as move ↑ toward P.

and to right ⟶ toward P.

Thermal heats of formation of cpds (Kcal/mol)

| | |
|---|---|
| $Cu_3P$ | -32. |
| $Fe_3P$ | -35.2. |
| $Ni_3P$ | -33. |
| | ↓ |
| $Ni_3Al$ | -37.6 |
| $NiAl$ | -34.0 |

phosphides are quite similar to aluminides in the respect.

PART NO. 990092

RUN NO. 3 DATE 10/15
OPERATOR: Berman
SAMPLE: Metco 95/5
ATM: Air @
FLOW RATE:

T-AXIS
SCALE, mv/in 0.8
PROG. RATE, °C/min 50
HEAT X COOL ISO
SHIFT, in 0

DTA-DSC
SCALE, °C/in
(mcal/sec)/in
WEIGHT, mg 72
REFERENCE $Al_2O_3$

TGA
SCALE, mg/in
SUPPRESSION, mg
WEIGHT, mg
TIME CONST., sec
dY, (mg/min)/in

TMA
SCALE, mils/in
MODE
SAMPLE SIZE
LOAD, g
dY, (10X), (mils/min)/in

BS=5.62/PA=20/AA=20
@ 350 → 1000°C

DU PONT instruments

MEASURED VARIABLE

PLAINTIFF'S EXHIBIT
IDN 11
12-6-10

RED

GREEN

100

0 100 200 300 400 500 600 700 800 900 1000 1100

TEMPERATURE, °C (Pt/Pt-13% Rh)

E1009

E1010

# POWDER METALLURGY

*Papers Presented at the 1940 and 1941 Conferences on Powder Metallurgy Held at the Massachusetts Institute of Technology, Cambridge*

Edited by

JOHN WULFF

Published by
The American Society for Metals
Cleveland, Ohio

1942

## II. Surface Coatings Produced From Metal Powders

The foregoing, the traditional and somewhat narrow field of powder metallurgy, concerned with molding and sintering of powders to produce finished parts, is not the only place in which metal powders are of service. There are also many other uses which are of great importance and interest.

**(A) Calorizing, Sherardizing and Other Metallic Cementation Processes** — Metal powders with or without a flux, when heated with clean iron or steel parts in sealed vessels, form an alloy bond integral with the surface of the work. With aluminum as the alloying metal, the finished piece is said to be calorized; with zinc, sherardized. These are the most important commercial operations, but other metals — notably silicon and chromium — can be similarly surface-alloyed with the work to confer on the baser metals the special properties of the cemented metal. Objects are cemented for a variety of reasons of which resistance to corrosion and heat oxidation are the most important.

**(B) Metal Spray Gun** — The Schoop gun has been used for spraying molten metal on various surfaces for a number of years. The sprayed metal should be in wire form. A more recently developed metal spray gun, the Schori, employs metal powders as a feed, and it is said that any metal available in powder form can be so sprayed. (Some metals are cheaper in powder form than in wire.) Zinc powder is the most used for such work, and also copper, aluminum, and lead powders are used in commercial quantities. Powders of many materials other than metal, such as plastics, glass, silicon, bitumen, and rubber, and, if desired, various mixtures of these with metal powders, can also be sprayed. Further developments in this field can be expected.

**(C) Paints** — While metallic pigment paints might be considered under this heading, they are of such great importance that they have been assigned a group classification of their own and will be treated below.

## III. Soldering and Brazing

**(A) Copper Brazing** — Copper powder is used to a very considerable extent for brazing steel parts. While copper wire and sheet are often used for this work, powder may be conveniently applied at and around the joint in the form of a paint or paste after being mixed with some liquid, such as petroleum spirits, alcohol, a lacquer, or water. The parts, thus coated with copper, are passed through a furnace in which a reducing atmosphere is maintained at a tempera-

E1012

# TREATISE ON POWDER METALLURGY

By

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## Volume III

*Classified and Annotated Bibliography*

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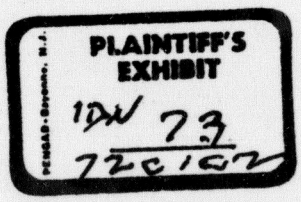


(5422) U. S. 2,482,423 (1949). V. T. Malcolm (The Chapman Valve Mfg. Co.), *Copper Base Alloy.* Powdered metallic Ti is placed in the bottom of a crucible and covered with a mixture of $KNO_3$, borax and ground charcoal. Then the crucible is charged with Cu-base metal, covered with a mixture of charcoal and borax, followed by melting in an oxidizing flame and de-oxidizing with a small amount of P. The Ti is approximately 0.10 to 1.25% of the alloy.

(5423) U. S. 2,486,433 (1949). T. S. Perrin and T. S. Seybert (Diamond Alkali Co.), *Production of Flux for Magnesium and Magnesium Alloys.* The flux is prepared by collecting waste from Mg electrolytic cells comprising sludge having 20 to 30% $MgCl_2$, 30% NaCl, 18 to 23% MgO, 6 to 15% Mg, 12 to 17% $CaCl_2$ and smaller amounts of $CaF_2$, Fe and graphite.

## v. Metal Surface Coating, Spraying, Metallizing, Cladding and Molding Compositions

(5424) U. S. 148,795 (1874). E. Wood (J. H. Legge), *Coating Iron and Steel to Prevent Oxidation.* A coating for rolled Fe or steel is composed of pulverized metals, *e.g.*, Pb, Zn, brass, Cu, Mn, Sn and Sb, borax, flake white and lime, and is fused and repowdered.

(5425) U. S. 242,649 (1881). W. H. Howes (Hale & Mulford, Inc.), *Method of Ornamenting the Surface of Jewelry.* To ornament metal buttons, solder filings are sprinkled over a layer of Au or Ag filings and the solder melted to fasten the Au or Ag.

(5426) U. S. 360,283 (1887). W. C. Edge, *Ornamenting Jewelry.* Cu wire is arranged on Au backings in a pattern, then Au or Ag filings are fused together around the Cu wire, which is dissolved.

(5427) U. S. 437,468 (1890). D. Wheeler (Acme Shear Co.), *Finishing Metallic Surfaces.* Handles of scissors are finished by coating them with Au size, applying the metal as powder and baking it.

(5428) U. S. 470,492 (1892). J. Kennedy, *Coating for Iron.* A coating for Fe comprises tobacco juice mixed with metal powders.

(5429) U. S. 546,446 (1894). G. Gross (Plymouth Cycle Mfg. Co.), *Coating of Bicycle Frames.* Bright steel tube bicycle frames are coated with size, baked to give the size tackiness, dusted with Al bronze powder, baked, varnished and baked again.

(5430) U. S. 629,426 (1899). G. D. Coleman, *Coating for Metal Parts.* A coating for metal ship bottoms consists of a layer of paint with a comminuted soft metal, second coating of paint, and a layer of comminuted Cu.

(5431) U. S. 639,537 (1899). W. C. Dickey, *Coating of Iron or Steel.* Sheet Fe and steel are protected by coating with a mixture of powdered Al and PbO, then hammering or rolling.

(5432) U. S. 682,173; 682,174 (1901). G. D. Coleman, *Protective Coating.* A protective coating for chemical holding tanks is applied by first coating with paint, then with comminuted metal or alloy, applying finely divided solder, and heating.

(5433) U. S. 682,914 (1901). G. D. Coleman, *Antifouling Coating.* A coating for metal ship bottoms consists of a layer of paint, a layer of finely divided vegetable insulation with powdered Cu hammered into coatings.

(5434) U. S. 717,080 (1901). G. D. Coleman, *Coating for Metals.* A corrosion- and high temperature-resistant coating for metal consists of an outer layer of powdered metal and an intermediate layer of comminuted soft metal.

(5435) U. S. 741,769 (1903). G. D. Coleman (Coleman Ship Coppering Co.), *Antifouling Coating.* A coating for metal ship bottoms consists of a layer of paint, a layer of ground, oiled, and Pb-coated wood and a layer of Cu powder.

(5436) U. S. 829,386 (1906). S. Cowper-Coles, *Deposition of Metal upon Metallic Articles.* Metal is coated with Sb in powdered form and heated.

(5437) U. S. 886,349 (1908). W. H. Connell, *Manufacture of Railway Rails.* A railway rail is given a track coating of pulverized Ni or Mn which is mechanically worked into the surface.

(5438) U. S. 896,751 (1908). E. Norton, *Composite Coated Steel Sheets.* Steel sheets are coated along the seams with varnish and Al bronze powder.

(5439) U. S. 904,444 (1908). J. W. Paton, *Coating Iron or Steel.* Finely divided metal is mixed with equal or greater quantity of powdered $SiO_2$ or C which is packed around the article, and the whole is heated.

(5440) U. S. 931,503 (1909). W. G. Scott (Chicago Varnish Co.), *Dipping Process for Coating Articles.* Articles are coated by dipping them into a bronze liquid bath containing bronze powder and blowing air through the bath for uniform coating.

(5441) U. S. 1,029,522 (1912). B. D. Avis, *Coating Aluminum.* For coating Al with Sn a composition of $SnCl_2$, $ZnCl_2$ and powdered Sn is used.

(5442) German 256,925 (1913). M. U. Schoop, *Method of Producing Metal Coatings.* Metal powder is centrifuged against the surface of the body.

(5443) German 280,752 (1914). W. A. Guertler, *Surfacing of Babbitt Metal.* Babbitt metal is coated with Fe by spraying finely divided Fe on the matrix of Pb.

(5444) U. S. 1,128,059 (1915). M. U. Schoop (Metals Coating Corp. of America), *Metallic Coatings.* Coherent metallic coatings are produced by projecting finely divided non-molten metal onto the metal surface by a reducing gas.

(5445) U. S. 1,161,944 (1915). J. H. Maddy and B. H. Schubert (The Lohmann Co.), *Protective Metal Coating.* Powdered Pb is mixed with an Hg salt and fused on frames.

(5446) U. S. 1,236,383 (1917). F. A. Fahrenwald (as represented by The United States of America), *Coating Tungsten.* W or Mo is coated with Au by applying powdered Au or an Au salt in a cleansing flux and heating.

(5447) U. S. 1,380,847 (1921). F. L. Soerenson (Metal Protection Labs.), *Treating Ferrous Articles.* Fe articles are provided with a corrosion resisting coating consisting of a mixture of caustic alkali, water, a metal powder and a metal-oxygen compound.

(5448) U. S. 1,431,395 (1922). H. G. Grinlinton, *Coating of Metals.* For tinning of Cu or brass, powdered Sn is mixed with a carrier, and stearic acid is used for a flux. The mixture is applied to the surface and heated until the Sn melts.

(5449) Brit. 225,072 (1924). H. Connelly, *Coating Composition for Metals.* The coating comprises an intimate mixture of powdered Sn, $CaSO_4$ and dextrin.

(5450) U. S. 1,565,495 (1925). C. W. Pfeil, *Protective Aluminum Coating.* A protective Al coating for Fe is made from powdered Al which is protected from oxide coating by adding oil.

(5451) U. S. 1,565,496 (1925). C. W. Pfeil, *Making Corrosion-Resisting Metals.* Sn-coated Fe metal is heated until the Sn melts; Al powder is applied until its saturation in Sn is reached.

(5452) German 437,502 (1926). V. Planer, *Coating of Wires.* The wire is passed through an emulsion of Sn dust in oil, and is heated after it emerges.

(5453) Brit. 261,017 (1927). E. D. Feldman, *Process of Coating Articles with Metal.* A sticky coating of bituminous substance is first applied and then atomized metal is applied with a Schoop gun.

(5454) German 469,358 (1928). R. Auerbach and W. Steinhorst, *Metal for Coatings by Spraying.* The metal powders are mixed with a binder and formed into wire.

(5455) U. S. 1,662,865 (1928). J. W. Powell, *Coating of Metal Articles.* A decorative coating for metal articles comprises a mixture of lacquer and Au-bronze.

(5456) U. S. 1,705,057 (1929). E. Brandus, *Telephone Cable Armor.* Telephone cable armor comprises a molded mixture of Fe powder and a finely divided Pb alloy.

(5457) U. S. 1,711,603 (1929). E. Lay, *Coating of Metal.* Al alloy powders of great uniformity for coating metals are obtained by adding Al to a powdered clay base in small amounts, igniting it in an inert atmosphere and repeating until the desired percentage of Al is obtained in the powder.

(5458) U. S. 1,729,065 (1929). W. H. Cole, *Material for Protecting Iron and Steel.* Fe or steel articles are coated by revolving them in a cylinder containing emery, Cu-Zn precipitate and Al powder.

(5459) U. S. 1,735,000 (1929). J. G. Dely (Chemical Research & Designing Co.), *Copper Coating.* To facilitate welding together of Cu particles used to coat steel or Fe plates, Cu particles are mixed with NaF.

(5460) Brit. 331,550 (1930). W. Baur, *Coating Metal Bodies.* Metal surfaces are coated with a varnish-like material containing metal powder.

(5461) U. S. 1,758,473 (1930). M. U. Schoop, *Coating Articles with Metal.* Metal coatings are produced by applying a reducing melting flame to a mixture of metal powders and a combustible liquid in a closed chamber.

(5462) U. S. 1,766,417 (1930). W. E. Watkins (Copper Plate Sheet & Tube Co.), *Forming a Film of a Plating Metal.* A film of a plating suspension in finely divided condition is formed on the metal to be plated by heating.

(5463) U. S. 1,804,991 (1931). G. A. Johnson (American Steel Pipe Co. Inc.), *Process for Coating Metal Surfaces.* Metal surfaces are coated by dipping into a bath containing metal powders and a liquid vehicle, including linseed oil, gum, and metal salts.

(5464) U. S. 1,810,409 (1931). W. E. Watkins (Copper Plate Sheet & Tube Co.), *Method of Plating Metals.* A plating alloy of two metals is applied on an Fe base by first plating with Zn, then applying Cu particles in a spreading medium, and heating.

(5465) Brit. 385,496 (1932). Telefunken Gesellschaft für Drahtlose Telegraphie m.b.H., *Coating Thermionic Valve Electrodes.* Powders of W, Mo, or Ta are used for the coating.

(5466) Brit. 402,203 (1933). British Refractories Research Assoc., *Producing a Coating of Oxide.* A powdered element, *e.g.* Al, Si, Ca, Mg, which forms on oxidation refractory oxides, is introduced into the air or the $O_2$ blast of an oxidizing flame which is directed upon the surface to be coated, to form a refractory coating on a metal base.

(5467) U. S. 1,893,830 (1933). R. P. Turner (New York Wire Cloth Co.), *Wire Fabric.* Wire fabric is produced by coating it with Al powder in a suitable vehicle, such as spar varnish.

(5468) U. S. 1,902,059 (1933). J. H. Critchett (Electro Metallurgical Co.), *Production of Alloy Surface Castings.* Fe-Cr surfaced castings are produced by lining molds with Fe-Cr powder.

(5469) U. S. 1,902,478 (1933). E. Wiegand (General Electric Co.), *Cathode.* A thermionic tube cathode is made by painting Ni in a volatizable vehicle onto porcelain tubing and then sintering the coating.

(5470) U. S. 1,939,667 (1933). H. Csanyi, *Metallic Coating.* A coating for use on a metallic base, *e.g.* Fe, is composed of a mixture of powdered Zn, Pb, Sn and a chloride dispersed in an aqueous solution; it is applied to the base by brushing or spraying and heated to fuse the coating. A typical composition is 45% Pb, 5% Sn, 39% Zn, and 10% $ZnCl_2$.

(5471) U. S. 1,953,508 (1934). O. Schober (American Lurgi Corp.), *Coloring and Protective Coating.* A coating and protective composition consists of a paint vehicle and a pigment of an Al-Si alloy; Si is present to increase corrosion resistance and heat transmission.

(5472) U. S. 1,980,670 (1934). H. A. Eckman and H. W. Maack (Crane Co.), *Inhibitor for Nitriding Process.* The inhibitor or the protective coating is made of 30-40% $NH_4$ phosphate in solution, 3-10% boric acid solution, 35-90% Sn powder, 5-60% ground $SiO_2$, 1-25% kaolin.

(5473) U. S. 1,998,506 (1935). W. S. Jones, *Mounting for Precious Metal in Leaf and Powdered Form.* The mounting consists of Al foil with a coating of metal powder and adhesive.

(5474) French 801,004 (1936). Drake, McGee and Hallstedt, Inc., *Coating Metal Surfaces.* A metal base surface, *e.g.* Fe, is coated with another metal, *e.g.*, Al, by applying to the base a mixture of finely divided Al and a volatile solvent, and heating to a temperature between the melting points of the two metals so that part of the Al diffuses into the Fe to form an intermediate layer.

(5475) U. S. 2,048,309 (1936). C. E. Williams, D. E. Krause and C. H. Lorig (The Battelle Memorial Institute), *Mold.* A mold for preparing enameled cast Fe for stoves is coated with Cu powder to produce a blister-free surface for the enamel.

(5476) Brit. 474,245 (1937). Remy, Van Der Zypen & Co., *Device for Metallizing Iron Bands or Wires. Cf.:* German 686,752 (No. 5484).

(5477) U. S. 2,072,229 (1937). L. Waitman, *Tinning Method.* Metal is tinned by dipping a wet sponge into a dry mixture of $SnCl_2$ and a tartrate, and then in pulverized Zn, and rubbing the sponge on the metal surface.

(5478) U. S. 2,078,808 (1937). J. V. Reardon and P. L. Goodale (Reardon Co.), *Metallizing Composition.* A dry powdered base for a metallic coating composition which produces a coating upon solution can be applied to steel and other structures.

(5479) U. S. 2,091,714 (1937). J. C. Matthews (Eastman Kodak Co.), *Metal Finish.* A protective coating for metallic mirrors comprises depolymerized chlorinated rubber containing a filler of metal powders.

(5480) U. S. 2,111,395 (1938). O. J. Hartwick (Pittsburgh Plate Glass Co.), *Coating of Steel Barrels.* A resin film for coating steel barrels contains metal powders as the stabilizing pigment.

(5481) U. S. 2,123,537 (1938). P. G. Marr, *Spraying Powders Such as Zinc or Aluminum on Steelwork.* An apparatus for spraying powders contains, between the main and base chambers, a constricted junction of the form of a Venturi tube.

(5482) U. S. 2,151,312 (1939). G. Ariotti (Atlas Powder Co.), *Hammered Metal Finish.* A hammered metal finish is produced on metal surfaces by applying, by a splattering operation, a coating composition comprising metal powder, a resinous base and a solvent.

(5483) U. S. 2,161,104 (1939). G. S. Smith, *Metallic Coating Suitable for Use on Iron.* A surface is coated by a mixture of Zn dust, $As_2O_3$, $TiO_2$ and $CrCl_2$.

(5484) German 686,752 (1940). Remy, Van Der Zypen & Co., *Device for Metallizing Iron Bands.* Fe bands are metallized by applying Al powder on a travelling lacquered Fe band which goes through a closed space; the device is equipped with brush rolls, and tubes through which air and metal dust are sucked out.

(5485) U. S. 2,254,013 (1941). R. Larson, *Hairpin.* A coating for hairpins comprises metal powder suspended in lacquer.

(5486) U. S. 2,262,072 (1941). T. H. Vaughn (Union Carbide and Carbon Research Laboratories), *Metallizing Operation.* Molten metal is sprayed onto an article to be coated, *e.g.* coating steel with bronze.

(5487) Canadian 436,644 (1942). Metallizing Engineering Co., *Process of Surfacing by Spraying Metal Powder.* The surface to be sprayed is roughened with Ni deposited from a wire electrode.

(5488) German 719,586 (1942). O. Kamps and H. Dettmann, *Process for Cladding Magnesium.* Mg or Mg alloys are dusted with Zn powder before cladding with Al to prevent formation of an Mg-Al alloy.

(5489) Swiss 219,342 (1942). M. U. Schoop, *Metal Coating.* The metal is heated in $N_2$ to produce a nitrided surface before it is sprayed with the metal powder used for coating.

(5490) Swiss 223,550 (1942). Metallisator Berlin A. G., *Spraying Gun.* Wire of a metal used for coating is pressed against the circumference of the friction gear with fixed axis and moves against the nozzle eccentrically to the axis of the nozzle system.

(5491) Swiss 224,135 (1942). M. U. Schoop, *Production of Metal Coatings.* The coating procedure consists of melting metal wires of W, Mo, Ta in the electric arc, and atomizing the melt by pressurized gas.

(5492) Swiss 226,459 (1942). M. U. Schoop, *Metallic Coatings*. The coatings are produced by spraying molten metal on the base metal with a compressed gas.

(5493) U. S. 2,279,268 (1942). L. V. Adams (General Electric Co.), *Coating for Metals*. A coating for metals comprises a mixture of Al powder and a Zn soap dispersed in a solvent.

(5494) U. S. 2,289,614 (1942). A. Wesley and H. Copson (International Nickel Co.), *Nickel Coated Ferrous Articles*. A Ni coating for Fe comprises Ni powder suspended in nitrocellulose lacquer or collolion.

(5495) French 882,372 (1943). Licentia, Patent-Verwaltungs G.m.b.H., *Coating of Electrodes*. The electrodes are coated with a paste of Zr or Ti, and then sprayed with a porous layer of the same metal.

(5496) Swiss 226,698 (1943). F. Gfeller and L. Baiker, *Spraying of Thermoplastic Materials*. Metal wire is agitated by the explosion of a combustible mixture, so that the separated particles are melted and thrown against the base.

(5497) Swiss 229,363 (1943). F. Gfeller and L. Baiker, *Apparatus for Metal Spraying*. Wire of a metal used for coating is preheated and fused, and is sprayed by several thousands of explosions per minute by a spray pistol equipped with an explosion chamber.

(5498) U. S. 2,308,704 (1943). C. J. MacNeil, *Instrument Illumination*. The pointer and dial of indicating instruments is coated with crushed Ag to make them visible against a blacked-out background.

(5499) U. S. 2,316,041 (1943). G. E. Armentrout (E. I. Du Pont De Nemours & Co.), *Process for Simulating Hammered Metals*. A simulated hammered metal finish effect, *e.g.* on metal surfaces, is produced by using a composition containing 1-5% flake metal powders, *e.g.*, Al bronze, and spattered with a solvent for the film-forming agent to produce a hammered effect.

(5500) U. S. 2,322,702 (1943). F. F. Peterson, *Shielded Cable*. A coating film of powdered Al is used on a high voltage shielded electric cable.

(5501) U. S. 2,326,001 (1943). G. Ariotti and E. H. Bucy (Atlas Powder Co.), *Simulated Hammered Material*. A simulated hammered metal finish on smooth metal or wood is produced by a coating containing powdered Al bronze and a lacquer; the partially dried coating is spattered with a volatile solvent for the base and of a definite viscosity, to dissolve some of the lacquer and thus cause a hammered effect.

(5502) U. S. 2,326,623 (1943). E. W. Crosby (E. I. Du Pont De Nemours & Co.), *Simulated Hammered Metal Effect*. A simulated hammered metal effect is produced by spraying on a surface, particularly of metal, a composition including a flake-metal pigment, *e.g.* Al.

(5503) U. S. 2,326,814; 2,326,815 (1943). D. C. Wobbe (American Can Co.), *Plate Cans*. Al paint is used as a coating for black plate cans.

(5504) U. S. 2,330,202 (1943). J. B. Brennan, *Electrode for Condensers*. A conductive and porous layer of Al is produced on electrodes by spraying finely divided molten Al metal on a metallic base and simultaneously directing a current of fluid against the base, to maintain the surface of the base at a temperature lower than the melting point of the sprayed metal.

(5505) U. S. 2,335,316 (1943). A. P. Shephard (Metallizing Engineering Corp.), *Apparatus for Conditioning Metal Surfaces*. An apparatus for conditioning metal surfaces by the application of metal coatings by spraying is described.

(5506) U. S. 2,338,713 (1944). S. Ewing, *Half Cell Electrode*. An electrode has a plating of sponge Cu on a metallic base.

(5507) U. S. 2,342,738 (1944). G. W. Jernstedt (Westinghouse Electric & Mfg. Co.), *Coating for Metals*. A corrosion-resistant coating compound for metal surfaces comprises a solution of Fe phosphate, free phosphoric acid, an oxidizing agent and powdered Zn or Cu.

(5508) U. S. 2,343,031 (1944). A. J. Spelker, *Stencil*. A metal stencil is used for imparting a design to the non-planar surface of a contoured article which has a conductive coating of powdered Fe.

(5509) U. S. 2,343,630 (1944). H. Atwood (Allen B. Du Mont Laboratories Inc.), *Shield for Cathode Ray Tubes.* The shield consists of particles of electrically conductive material of high magnetic permeability, such as pure Fe, applied in an adhesive binder to the tube.

(5510) U. S. 2,348,912 (1944). E. H. Land (Polaroid Corp.), *Producing Designs.* Designs are produced in light-polarizing areas having a metallic backing plate with a reflecting surface of Al flakes.

(5511) U. S. 2,351,940 (1944). J. Dupuis, *Electroplating of Articles.* Metallic articles with a lacquer film containing lithopone and Cu powder are electroplated.

(5512) U. S. 2,350,583 (1944). L. N. Hampton (Bell Telephone Laboratories), *Heat Transferring Element.* A heat transferring element for electrically heated soldering tool has a tapering portion of Cu, coated with powdered Fe and Al.

(5513) U. S. 2,358,171 (1944). L. B. Lindemuth (Steel Ingot Production, Inc.), *Ingot Mold.* A cast Fe mold has a crevice between the tapered circular portions of the mold and the "stool" (cast Fe base) filled with metal powders, *e.g.* Fe-Si, Fe-Cr, Fe-Mn, to prevent the molten metal seeping out between the mold and "stool."

(5514) U. S. 2,359,436 (1944). J. J. Mascuch (Breeze Corp. Inc.), *Metallic Shield.* Spark plug conductors are electroplated with metal powders.

(5515) U. S. 2,361,897 (1944). I. E. de Graaf (Hartford National Bank & Trust Co.), *Coated Bearings for Vacuum Vessel.* Ball or roller bearings for high vacuum vessels have a coating formed of Pb powder, which prevents cutting, friction and the like.

(5516) Brit. 569,784 (1945). M. Cook, G. Parker and Imperial Chemical Industries, Ltd., *Clad Metals.* Alloys of Cu and Ag are cladded onto a base metal by sandwiching a thin foil of P-Cu between the cladding alloy and base metal, heating to 645-780° C. (1190-1430° F.) and pressing to consolidate. Alternately, the P-Cu may be formed into wire and sprayed on with a spraying pistol.

(5517) Brit. 572,103 (1945). W. S. Murray, *Wear Resisting Surfaces for Bearings.* Pb is alloyed with In and used as a film of 0.001-0.03 in. thickness; it is sprayed directly on a steel or Fe backing.

(5518) U. S. 2,366,850 (1945). M. G. Gardner (Pittsburgh Plate Glass Co.), *Lining for Food Containers.* A composition for use as liner for metallic food containers contains flake Al powder.

(5519) U. S. 2,372,124 (1945). E. S. Schenkel, *Sign Construction.* An illuminated metal sign utilizes Al powder in a light diffusing coating to reflect the light.

(5520) U. S. 2,378,588 (1945). J. W. Skehan and G. I. Agule (Machlett Laboratories Inc.), *Method of Making Bearings.* Ball bearings utilize Ag particles as a coating on the working surfaces.

(5521) U. S. 2,380,047 (1945). F. W. Hyman, *Apparatus for Applying Coatings.* An apparatus for applying coatings of metal powders to rough surfaced materials of metals is described.

(5522) U. S. 2,389,702 (1945). V. G. Ullmer (Haynes Stellite Co.), *Apparatus for Treating Metal Articles.* An apparatus for coating metal articles with metal powders comprises a blow pipe, having a nozzle with a passage for powdered metals, and a passage for an oxy-fuel gas mixture.

(5523) U. S. 2,390,183 (1945). R. A. L. Seligman, *Sheet Metal Die.* Steel sheets containing the working face of the die are welded together, and the back side is built up with a mixture of metallic particles, containing one metal whose melting point is not higher than the temperature at which the hardening of the steel face plates can be accomplished; the processing is completed by heating and compressing together the face plates and the metallic mixture.

(5524) Brit. 577,979 (1946). Imperial Chemical Industries, Ltd. and J. L. Coltman, *Heat Exchange Device.* A paste of Ag powder suspended in collodion is used for thinly coating thin-walled Cu or Cu-base elements.

(5525) Brit. 578,455 (1946). Metallizing Engineering Co., *Spray Gun for Gas-Blast Spraying.* A gun for spraying powdered metals or plastics on a metal wire is described.

(5526) U. S. 2,392,879 (1946). T. J. Radcliffe (General Electric Corp.), *Coating of Electrode.* One side of an elongated metal strip is coated with a suspension of powdered antiglow metal in a volatizable binder; the coating is baked on electrically.

(5527) U. S. 2,394,065 (1946). G. W. Jernstedt and J. C. Lum (Westinghouse Electric Corp.), *Protective Coating.* Powdered Fe is used in a protective coating for nonferrous metals.

(5528) U. S. 2,408,515 (1946). A. G. Hopkins, *Coating Process.* An Sn lacquer, comprising powdered Sn held in suspension by a vinyl-resin binder, is applied to black Fe surfaces.

(5529) U. S. 2,409,514 (1946). G. W. Pratt (Radio Corp. of America), *Cathode Ray Tube.* Powdered metal coatings are used for the inner wall of a cathode ray tube.

(5530) U. S. 2,409,769 (1946). W. E. Leyshon (Sylvania Products Inc.), *Fluorescent Glow Lamp.* Powdered Zr, suspended in nitrocellulose and amyl acetate, is used as an inner coating for the electrode in fluorescent glow lamps.

(5531) U. S. 2,412,201 (1946). J. B. Brennan, *Electric Device.* Al particles are sprayed onto a wire screen to make an electrode for condensers or lightning arresters.

(5532) U. S. 2,412,528 (1946). J. C. Morrell, *Coated Metal Container.* Metal powders are used in coating tin cans.

(5533) Brit. 589,645 (1947). C. F. Lumb, *Applying Coating by Spraying.* Metal powders are flame-sprayed on a metallic surface which first has been given a flame-sprayed plastic coating.

(5534) Brit. 589,807 (1947). A. F. Berry, *Heating Apparatus for Cooking Purposes.* Powdered cast Fe or Ni is sprinkled onto enamel-coated electric hotplates made of cellular refractory material.

(5535) Canadian 442,455 (1947). Federal Telephone & Radio Corp. and M.C. Bloom, *Coating with Selenium. Cf.:* U. S. 2,426,173 (No. 5540).

(5536) U. S. 2,414,510 (1947). E. A. Doyle (Linde Air Products Co.), *Apparatus for Cladding Composite Metal Bodies.* The cladding process involves preheating the Fe metal body, cleaning the surface by a thermochemical desurfacing operation to remove a layer of the metal, *e.g.* by $O_2$ and a heating flame, applying the cladding material in form of a spray or powder, and compressing the composite body between rolls.

(5537) U. S. 2,414,511 (1947). H. H. Dyar (Linde Air Products Co.), *Apparatus for Cladding Metal Bodies.* The cladding is applied by subjecting the surfaces to be joined to a skinning or thermochemical desurfacing operation with $O_2$, applying a metal powder and a flux, and pressing the metals together.

(5538) U. S. 2,414,923 (1947). C. Batcheller, *Metal Cladding by Spraying.* Powdered metal is sprayed on a surface, so prepared that oxidation is avoided; high pressure is used to compress the sprayed particles into a dense, smooth surfaced mass.

(5539) U. S. 2,421,154 (1947). C. E. Maier (Continental Can Co. Inc.), *Aluminum Powder and Resin Spot Facing Material.* A flexible closure liner facing material for beer containers consists of Al flake powder distributed throughout the resin material, whereby the aluminum flakes are enclosed in the resin, to reduce the rate of permeation of moisture or gas.

(5540) U. S. 2,426,173 (1947). M. C. Bloom (Federal Telephone & Radio Corp.), *Coating with Selenium.* An electrically conductive base element such as C is treated with an alkaline selenide solution, such as an $Na_2Se$ solution which is prepared by passing $H_2Se$ through a solution of NaOH.

(5541) Brit. 596,687 (1948). The M-O Valve Co. Ltd. and R. O. Jenkins, *Thermionic Valve.* Valve grids are coated with a suspension of a 75:25 Mg-Ba alloy powder.

(5542) Brit. 593,653 (1948). Callite Tungsten Corp., *Coating of Metals with Metals and Alloys.* The metal or alloy which is to form the coating is chosen

partly from the metal powder and partly from a powdered compound, *e.g.* the oxide or nitrate; this mixture is suspended in an organic solution of nitrocellulose. Wires are passed through the suspension and then through the sintering furnace.

(5543) Brit. 602,530 (1948). Bristol Aeroplane Co. Ltd. and F. M. Owner, *Gas Turbine Blades.* A turbine blade designed for air or liquid cooling is given a coating of sintered heat-resistant ceramic material.

(5544) Brit. 603,553 (1948). Schori Metallising Process Ltd. and C. F. Lumb, *Coating Surfaces.* The metal can be flame-sprayed with paraffin wax or shellac wax which may contain a filler, such as $SiO_2$ or Zn dust.

(5545) Brit. 604,457 (1948). C. F. Lumb, *Production of Heat Resistant Coatings.* Metallic articles are first flame-sprayed with Al powder, then flame-sprayed with a coating of mixed Ni-Cr-Al, and finally heat treated.

(5546) Brit. Appl. 13310/48 (1948). Charles Hardy Ltd. and W. N. Pratt, *Metal Powder Product.* Flame-spraying of metal powders which form highly refractory oxide films on passing through the oxidizing zone of the flame, *e.g.* stainless steel, alloys of Cr or Al, is facilitated by coating the particles with a silicone resin.

(5547) Brit. Appl. 13311/48 (1948). Charles Hardy, Ltd. and W. N. Pratt, *Aluminum Steel-Backed Bearings.* Steel strip is heated to 480° C. (896° F.), and a molten layer of Al is built up by flame-spraying the powder with a neutral or reducing flame, using as carrier gas He, mixed with $N_2$ or $CO_2$.

(5548) French 937,824 (1948). Industrial Metal Protectives, Inc., *Protective Coating for Metallic Surfaces.* Zn powder is suspended in water glass having an $Na_2O{:}SiO_2$ ratio of 1:2.6.

(5549) French 944,627; 944,628; 947,479 (1948). G. Bourdillion, *Pistol for Metallizing by Means of Powders.* The arrangement of the ducts and the powder container in a flame-spraying pistol is described; the pistol may be used for sand-blasting.

(5550) U. S. 2,434,855 (1948). J. L. T. Kosterlitzky, *Coating Metal Articles.* An article containing Cu is rubbed with a mixture of 22 parts of $FeCl_2$ and $SnCl_2$, 10 parts powdered Zn, 20 parts K bitartrate, 9 parts $NH_4Cl$, 20 parts $NiSO_4$, 55 parts Ni ammonium sulfate, and 6 parts NaCl.

(5551) U. S. 2,437,612 (1948). H. Osborg, *Plating Magnesium Electrolytically with Zinc.* The process entails using a specially treated cyanide bath with an Mg test cathode to deposit a continuous adherent coating of Zn.

(5552) U. S. 2,438,205 (1948). J. E. Coates (Douglas Aircraft Co. Inc.), *Measuring Instrument.* The device is manufactured by depositing a film of conductive material upon the body in form of a grid of the desired shape by means of a stencil, formed with an opening of the shape of the grid, and depositing through the opening a metallic coating of Cu or bronze onto the body.

(5553) U. S. 2,438,967 (1948). H. D. Ellsworth (Indium Corp. of America), *Indium-Gold Surfaced Article.* The coating comprises a surface film with 10% In in the alloy, an underlayer of Au containing 8% In and an intermediate layer in which the proportion of In increases towards the surface.

(5554) U. S. 2,440,969 (1948). V. C. Nightingall, *Protective Coating for Ferrous Metal Surfaces.* A hard protective coating on Fe metal surfaces is produced by applying a composition of Zn and Pb powders, which are incorporated in an aqueous solution of alkali silicate and $NaHCO_3$ and drying the applied coating.

(5555) U. S. 2,442,485 (1948). F. C. Cook, *Method of Coating Hot Rolled Ferrous Products.* The products are coated by passing the hot bodies from the deoxidizing gas chamber into the coating chamber filled with vapor of the coating metal, while controlling the temperature of the bodies in the coating chamber, to condense the coating metal on the spongy surface of the bodies, causing it first to alloy and then forming the coating.

(5556) U. S. 2,444,422 (1948). V. H. Bradford (Specialities Development Corp.), *Aluminum Coating for Steel.* The Fe base is subjected to nascent N; the nitrided base is then coated with Al by spraying and then passed into a furnace.

(5557) U. S. 2,444,914 (1948). J. B. Brennan (Everett D. McCurdy), *Electrolytic Device*. The device comprises a thin sheet of metal having pierced openings for the flow of the ions; the openings are surrounded by minute projections composed of the displaced material, and both sides of the sheet are sprayed with metal particles.

(5558) U. S. 2,446,387 (1948). T. F. Peterson, *Shielded Cable*. A cable which has a surface conductivity of 10,000 ohms per inch is coated with a lacquer of a flexible oil-extended phenol-aldehyde resin in a volatile solvent containing dispersed metal powder.

(5559) U. S. 2,450,850 (1948). W. H. Colbert and A. R. Weinrich, *Coating by Evaporation of Metals*. The coating is applied by evaporating a metal, *e.g.* Cu, (as an alloy with Ca) from a filament of W, Mo, Ta; heating causes the metal to wet and to spread out over the surface of the filament, and to deposit a coating on a polished support material.

(5560) U. S. 2,454,910 (1948). W. P. Carr (De Vilbiss Co.), *Method of Applying Coatings*. Surfacing of a molded article is effected by coating the mold surface with an adhesive of non-permanent nature, by partially embedding in it inert particles, such as Al flakes, Au bronze or abrasive material, and filling the mold with the molding material; after hardening, the article is withdrawn, and the adhesive is removed, leaving the inert particles fixed in the molded article.

(5561) Brit. 609,644 (1949). Schori Metallising Process Ltd. and C. F. Lumb, *Method of Coating Metal Surfaces with Metals*. Improved results are obtained by flame-spraying metal powder mixed with a small amount of a plastic material for pore filling; *e.g.*, steel sheet is sprayed with Zn powder mixed with 3% polyethythene.

(5562) Brit. 621,244 (1949). Schori Metallising Process Ltd. and W. D. Jones, *Coating of Surfaces*. Corrosion-resistant coatings on Fe or steel are obtained by flame-spraying an Al alloy powder with 30-40% Mg or 10-20% Zn.

(5563) Brit. 623,212 (1949). Chemal Trust, *Apparatus for Deposition of Aluminum Coating*. The coating is built up by repeated applications of alternated Al paste and Al powder to both sides of Fe sheets; each application is followed by sintering and equalizing the layers.

(5564) Brit. 624,066 (1949). N. V. Enkes, *Atomizer for Discharging Molten Material*. In a pistol used for flame-spraying metal powders, ducts which are exposed to wear are made of WC.

(5565) Brit. Appl. 2631/49 (1949). Radio Corp. of America, *Non-Alloying Zirconium Coating Material*. Zr coatings, on anodes of Fe or Mo in electron discharge tubes, are prevented from alloying with the base by diluting Zr hydride of Zr metal powder with pure $ZrO_2$.

(5566) Swiss 255,979 (1949). A. Jenny, *Heat-Resistant Surfaces on Iron and Steel*. The coatings are produced by spraying with Al or Al alloys at 250° C. (480° F.) so that part of the coating alloys with the base metal.

(5567) U. S. 2,457,806 (1949). E. R. Crippa, *Inductance Coil*. The coil has a coating comprising a wax or resin binder and particles of finely divided Fe.

(5568) U. S. 2,458,839 (1949). J. R. Dyer and T. J. Rowan (The Indium Corp. of America), *Electrodeposition of Indium and Its Alloys*. The bath compounds may be made up in powder formed and dissolved in water at the time of use; in one example, a flat anode of Pb and In powder compressed into plate form is used.

(5569) U. S. 2,461,556 (1949). E. T. Lorig (Carnegie-Illinois Steel Corp.), *Method and Apparatus for The Electrolytic Coating of Metal Strip*. The anode may assume the form of a meshlike body in which filaments of plating metal are interwoven with each other for use in this form, or as a carrier strip upon which the plating metal may be deposited in powdered or granular form and sintered or otherwise fixed upon the backing.

(5570) U. S. 2,462,157 (1949). W. E. Blackburn (Westinghouse Electric Corp.), *Method of Eliminating Porosity in Crystalline Selenium Films*. After forming the Se coating, the coated plate is immersed in a solution of NaOH in glycerine; after annealing, the Se surface is Schoop sprayed with an alloy comprising 75% Sn and 25% Cd.

(5571) U. S. 2,462,681 (1949). V. J. Schaefer (General Electric Co.), *Method of Forming Germanium Films.* A method of forming a coating of Ge on a solid surface comprises bringing the reducing portion of a low velocity gas flame (from a Bunsen burner) into contact with a mass of powdered Ge oxide and bringing the tip of the flame into contact with the surface to be coated.

(5572) U. S. 2,462,763 (1949). V. C. J. Nightingall (Di-Met Proprietary Ltd.), *Protectively Coated Ferrous Metal Surfaces.* A method involves applying to the Fe surface a coating of a composition comprising finely divided metallic Zn incorporated in an aqueous solution of an alkali silicate, and converting the coating to an insoluble condition by reacting it with $CO_2$.

(5573) U. S. 2,463,342 (1949). S. B. Wiczer, *Metallic Coatings.* A hot-rolled Mg sheet is treated with Al or Mg metal powder and an oxide of a metal which will slow down oxidation, such as $B_2O_3$, and another oxide tending to form a slight coating, such as BeO.

(5574) U. S. 2,472,760 (1949). H. L. Ratchford (Sylvania Electric Products Inc.), *Electrode for Electron Discharge Devices.* The wire grid electrode comprises an alloy of approximately 96% Pt and 4% W and is sprayed with a lacquer suspension of W powder of approximately 325 mesh and is then mounted in conventional manner.

(5575) U. S. 2,475,601 (1949). A. O. Fink (The Commonwealth Engineering Co. of Ohio), *Bonding of Metal Carbonyl Deposits.* A Cu base is coated with Ni by first being exposed to a $CO_2$ atmosphere containing 1.4% by volume of $Ni(CO)_4$ for about 2 minutes while the Cu base is heated to 190° C. (375° F.); the coated base is then heated for about 15 min. in an atmosphere of natural gas and is then exposed to a $Ni(CO)_4$-containing gas until a coating of the desired thickness is obtained.

(5576) U. S. 2,485,176 (1949). F. D. Waterfall (Imperial Chemical Industries Ltd.), *Selective Carburization of Metals.* A stopping-off composition for use in selective carburization comprises a solution containing a volatile solvent of a lacquer which dries to a hard film and has suspended therein a metal powder formed of Cu mixed or alloyed with up to 15% Sn, together with a minor proportion of $Pb_3O_4$.

## vi. Cementation and Impregnation

(5577) U. S. 701,298 (1901). S. Cowper-Coles (Sherardizing Syndicate, Ltd.), *Process of Depositing Metals on Metallic Surfaces by Cementation.* Zn is deposited on a metallic surface by applying pulverulent Zn to the surface and heating.

(5578) U. S. 910,369 (1909). F. W. Gauntlett (Sherardizing Syndicate Ltd.), *Depositing Metals upon Metal Articles by Cementation.* To coat metal articles, finely divided metal is mixed with powdered $SiO_2$ or C, packed around the article, and the whole is heated.

(5579) U. S. 948,663 (1910). A. Sang, *Coating Metals by Cementation.* Fe or steel articles are coated by immersing them in Cu or Zn dust and passing electric current through them.

(5580) U. S. 986,504 (1911). A. J. Rossi (Titanium Alloys Manufacturing Co.), *Process for Treating Metal Bodies.* To coat steel with Ti, a powdered alloy of Ti and Fe is packed against the steel body and heated.

(5581) U. S. 1,014,749; 1,014,750 (1912). C. F. Burgess (United States Sherardizing Co.), *Coating Metal Parts with Metal.* To coat Fe or steel articles, Zn-Fe powder in granular form is applied to the surface and the whole heated.

(5582) U. S. 1,091,057 (1914). E. G. Gilson (General Electric Co.), *Process of Treating Metals.* To protect metals against corrosion they are heated in a non-oxidizing atmosphere with a powdered mixture of pure Al, $Al_2O_3$ and a chloride.

(5583) U. S. 1,155,974 (1915). T. Van Aller (General Electric Co.), *Treatment of Metals.* To protect Fe or Cu bodies from oxidation they are heated with Al powder to alloy the Al with the surface.

# ERNEST F. FULLAM, INC.

## SCIENTIFIC CONSULTANTS









E1023

*Scanning Electron Microscopy*

*Transmission Electron Microscopy*

*Electron Microprobe Analysis*

*Electron and X-ray Diffraction*

# ERNEST F. FULLAM, INC. / SCIENTIFIC CONSULTANTS

*Light Microscopy*

*Prototype Instrument Development*

E1024

E1025

Modern scientific investigation often requires the assistance of an organization with the widest possible experience in a particular field.

Working with a unique combination of instruments, the members of our staff specialize in microanalysis. Our ideal laboratory environment permits costly equipment to yield its fullest capabilities in helping to solve difficult research and development problems.

In addition, simpler "trouble-shooting" tasks are often performed here, either because the client lacks the necessary equipment or because of work load limitations in his own laboratory.

Ernest F. Fullam, Inc. was created for the specific purpose of providing prompt service involving the coordinated application of microscopy, diffraction, and microprobe analysis to all fields of industrial investigation.

Ernest F. Fullam

*President*

E1026

# THE COMPANY

For many years Ernest F. Fullam, Inc. has specialized in the analysis of materials through microscopy and related techniques.

Early to recognize the limitations as well as the advantages of the light microscope, certain members of the staff were among the first to visualize the wide range of scientific fields which could be extended by the use of the electron microscope.

The corporation was organized in 1953, and in 1958 the company moved to its new laboratory specially designed to permit the most effective utilization of its unusual complement of equipment and talents. By developing new and improved techniques for sample preparation, we have enabled the electron microscope to realize its full potential as a most valuable instrument for industrial research. When the X-ray microscope became available, it was utilized to reveal further details not visible by other means. In due course electron and X-ray diffraction apparatus was added to our facilities.

To broaden the available choice of methods for handling problems and interpreting results, an electron microprobe was designed, built, and put into service in our laboratory. Then, recognizing the customer's desire and ability to analyse his material, unaltered in the examination, the scanning electron microscope was added to our facilities.

Such pioneering sets Ernest F. Fullam, Inc. apart as a unique organization dedicated to continuous progress in its specialized field.

INVESTIGATIONS AND NON-DESTRUCTIVE ANALYSES conducted by Ernest F. Fullam, Inc. cover a wide range of industrial applications. Over the past years the laboratory has helped to solve problems involving over a hundred different materials. Among them:

Abrasives
Absorbents
Activators
Aerosols
Aggregates
Air Contaminants
Amorphous and Crystalline Particulate Materials
Apertures
Armature Surfaces
Barrier Materials
Bearings
Bonding Agents
Brake Shoes
Brushes
Carbon Black
Carbon Brushes
Carbon Papers
Catalysts
Cements
Ceramics
Cigarettes
Clays
Coating Materials
Colloids
Contact Surfaces
Contaminants
Corrosion Products
Diatoms
Dielectrics
Diffraction Grating Replicas
Dispersions
Driers
Dusts
Dyes
Electrodes
Emulsions
Face Powders
Felts
Ferrites
Fibers
Fiberglas
Filaments
Fracture Surfaces
Filter Media
Fluorescent Materials
Fumes
Glass
Greases
Hairs
Inclusions
Industrial Finishes
Inks
Ink Films
Insecticides
Insulating Materials
Integrated Circuits
Interfaces
Lakes
Lapping Materials
Latex
Leather
Magnetic Materials
Magnetic Tapes
Monolayers
Metals
Meteoric Debris
Micrometeorites
Minerals
Natural Fibers
Nucleating Materials
Oil Cloth
Oils
Oxides
Paint Films
Paints
Paper
Paper Fillers
Phosphors
Photographic Materials
Pigments
Plastics
Polarizing Materials
Polishes
Powdered Metals
Printing Inks
Printing Plates
Radioactive Materials
Rectifiers
Refractory Materials
Resins
Rocks
Rubber
Screens
Semi-conductors
Silicates
Silicones
Smokes
Soaps
Steel Alloys
Tars
Textiles
Thin Films
Tobacco
Transistors
Vehicles
Water Contaminants
Wood Fillers
Wood Products
Yarns

E1027

# WHAT THE COMPANY CAN DO FOR YOU

**Consulting services are offered in:**

Physical metallurgy, fractography and failure analysis
High altitude particle research
Integrated circuit structural and failure analysis
Biological pollution source detection
Atmospheric pollution identification
Corrosion studies
Material contamination
Surface studies
Patent infringement
Particle size analysis
Colloid and emulsion studies
Crystallography
Legal testimony
Prototype instrument development
Microscopy of organic and inorganic materials

**To meet your particular needs for problem solution our laboratory techniques include:**

electron microscopy by transmission and scanning . . . X-ray and light microscopy . . . electron microprobe analysis . . . electron and X-ray diffraction . . . chemical microscopy . . . photomicrography . . . ciné and color micrography . . . stereoscopic micrography . . . contour plotting . . . microrefractive index . . . and density measurements . . . areal analysis . . . thickness measurements . . . thin cross-sectioning . . . ultramicroscopy . . . vacuum evaporation . . . thin film deposition . . . shadow casting . . . dispersions . . . projection and contact micro radiography . . . micro X-ray emission . . . "clean room" services . . . preparation and loading of substrates for high altitude particle collection . . .

E1028

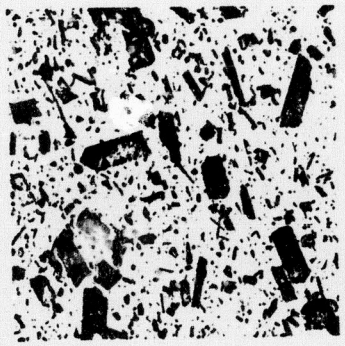
# TRANSMISSION ELECTRON MICROSCOPY - TEM

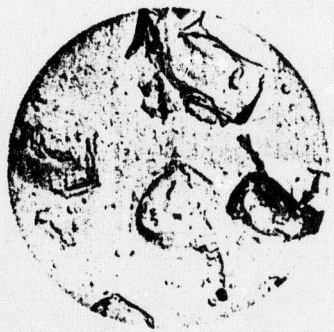
The transmission electron microscope has over 100 times the resolving power of the light microscope. Our microscopes provide direct magnification of more than 170,000 diameters. Photographic enlargement to over 1,000,000 diameters shows an image resolution of better than 4Å point to point.

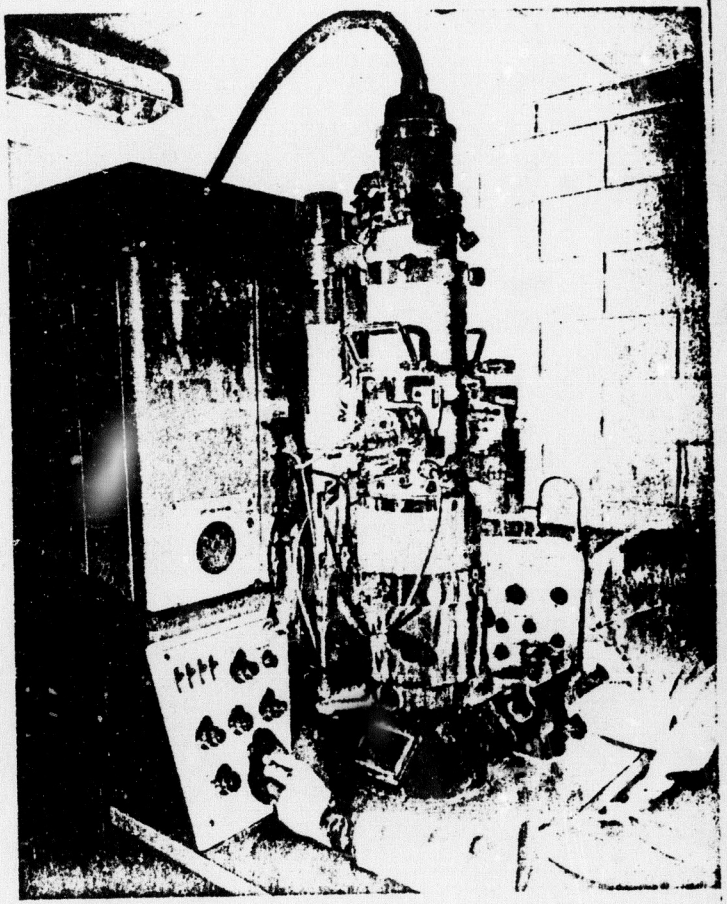
Acceleration voltages are 30 to 120 KV depending on desired image contrast and specimen thickness. Various accessories permit the specimen to be heated, cooled, tilted to high angles and rotated while being examined in either bright or dark field. Sample damage is prevented by a device that permits very low beam intensities to be used.

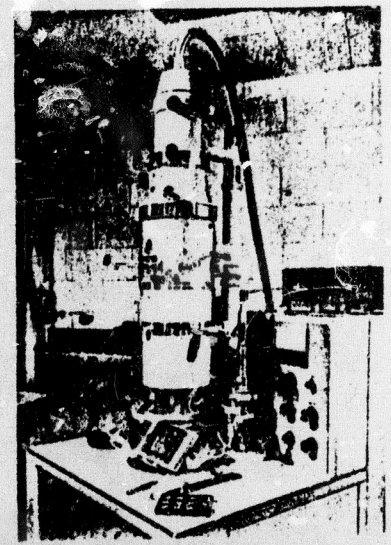
Stereoscopic studies and contour plotting can be performed. Crystalline compounds can be analyzed either by selected area and high resolution electron diffraction of transmission samples or by reflection diffraction of bulk samples.

The transmission electron microscope is an extremely versatile instrument with significant applications in all fields of scientific investigation.









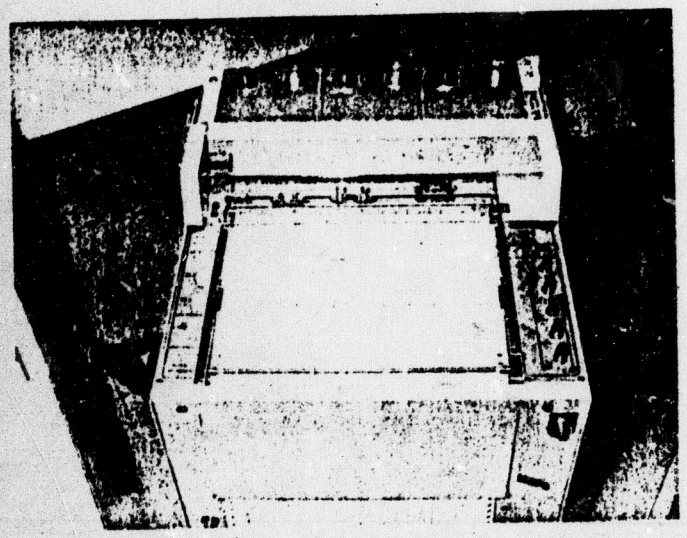
E1029

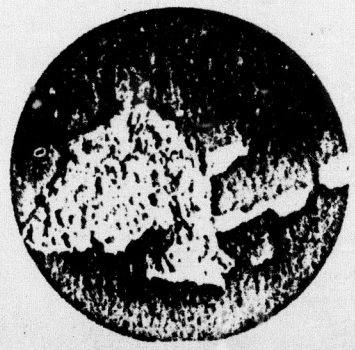
# ELECTRON MICROPROBE ANALYSIS

The electron microprobe analyzer is one of the most powerful and sophisticated tools for research, development and production problem solving. It provides precise non-destructive chemical analysis — both quantitative and qualitative — of minute volumes of solid materials.

Signals from its detectors are read out from solid state devices on to a strip chart, scaler, teletypewriter or punched tape for subsequent quantitative computer analyses. Visual display on a cathode ray tube enables photographic recording of X-ray, specimen current and back scattered electron images.

Applications of the electron microprobe are outstanding in the fields of metallurgy, chemistry and biology to study diffusion, corrosion, segregation, inclusions and particulates.





E1030

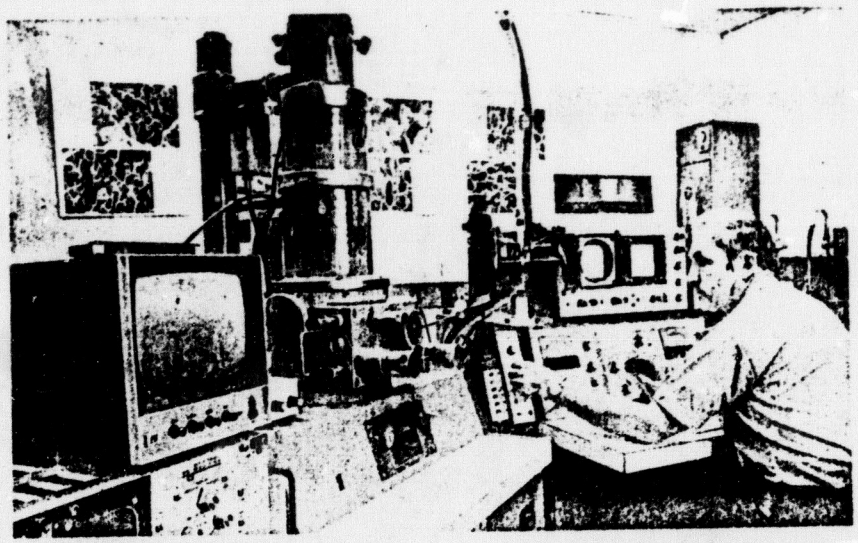
# SCANNING ELECTRON MICROSCOPY-SEM

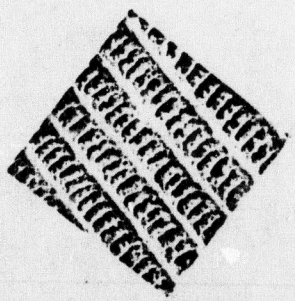
Scanning electron microscopy is an indispensable adjunct to light microscopy and transmission electron microscopy for the microstructural examination of a broad spectrum of materials in many forms.

The SEM is an imaging microscope for viewing surfaces at a wide range of magnifications on a CRT or television screen. With its energy dispersed X-ray read-out, the SEM identifies elements and provides qualitative and semi-quantitative analysis. A line graph of X-ray intensity superimposed on a photomicrograph will identify elements in the area of interest.

The special utility of the SEM stems from a unique combination of features:

- Great depth of focus—nearly 300 times that of the light microscope
- Variable specimen illumination and choice of electron imaging mechanism for realistic "three dimensional" imaging
- Wide magnification range—continuously variable — 50 to 100,000 diameters
- Good resolution—normally 200Å, potentially 100Å point to point
- Direct examination without replication or surface preparation
- Specimen manipulation with a micro manipulator and other special stages





Scanning electron microscopy is particularly useful for:

- Fractography of metals, ceramics, plastics, and composite materials
- Studies of pore and cell structures in natural and synthetic substances
- Determination of the topography of nearly any solid surface
- Evaluations of the particle size, shape and surface structure of powders, pigments, fillers, tissue cells, bacteria and other particulate matter
- Plating thickness measurement
- Investigations of coatings, deposits, corrosion layers, and biological tissue
- Examination of filament and fiber configuration and bonding in textiles, yarns, felts, papers, filters, wood
- Inspection of physical characteristics of semi-conductors surfaces and detection of potentials in transistors and integrated circuits.

Attendance by the client during scanning electron microscope examination of his specimens is cordially invited.

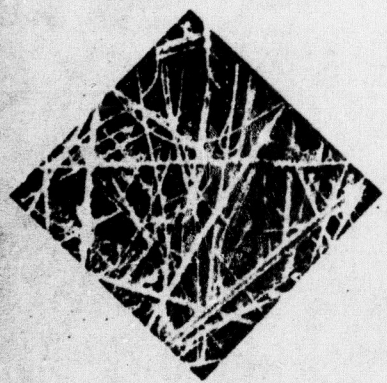
E1031

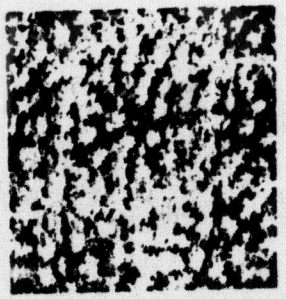
# LIGHT MICROSCOPY

The light microscope is the basic tool for the study of all materials in the field of microscopy and for the preparation of samples for study in other types of microscopes, diffraction instruments, and the electron microprobe.

Various types of light microscopes are available in our laboratory, including metallurgical, polarizing, stereoscopic, petrographic, photomacrographic, etc. Photomicrographs in black-and-white and color may be made of virtually all materials, many of which cannot be examined in other microscopes.

Numerous accessories and lens combinations give the light microscope a distinct advantage over both electron and X-ray microscopes in the study of many materials.





# X-RAY MICROSCOPY

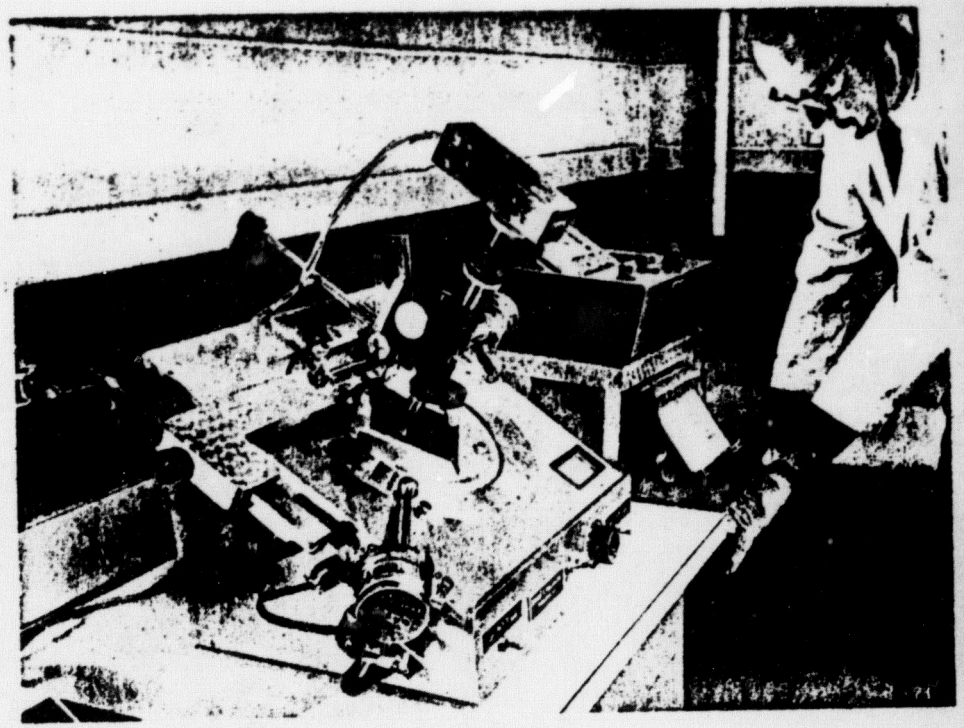
The X-ray Microscope is an electrostatically focused electron optical instrument producing X-rays for shadow projection in order to magnify and reveal detailed internal structures of specimens opaque to light and electrons. Among its many advantages are: good resolving power, greater penetration and very large depth of field permitting stereoscopic and ciné micrography. The very small source of X-rays, produced by this microscope, has been found useful for many research and analytical applications.

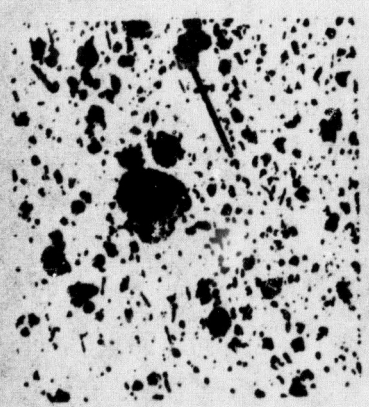
# ELECTRON DIFFRACTION

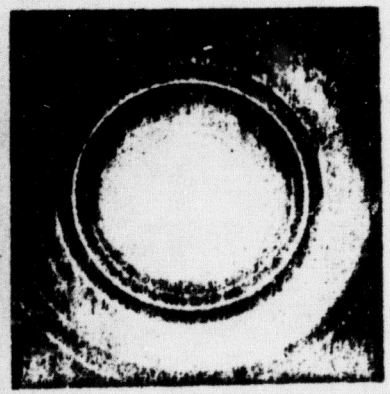
The electron beam formed in the electron diffraction camera, having a low penetrating power, produces diffraction patterns from the surface structures of bulk samples by reflection. Patterns may also be formed by electron transmission through thin films or small particulate materials which cannot be "seen" by X-rays.

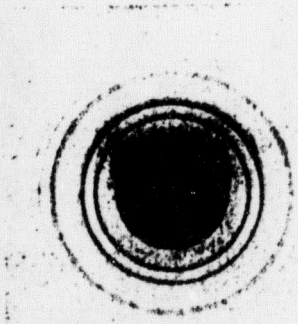
Chemical composition and crystalline structure of the material is then determined by measuring the spacings of spots or rings in the electron diffraction patterns.

Electron diffraction is a non-destructive analytical technique used on those materials or surfaces that do not produce an X-ray diffraction pattern for the identification of compounds. It is the only method capable of investigating structural features of thin films on solid matrices.









E1033

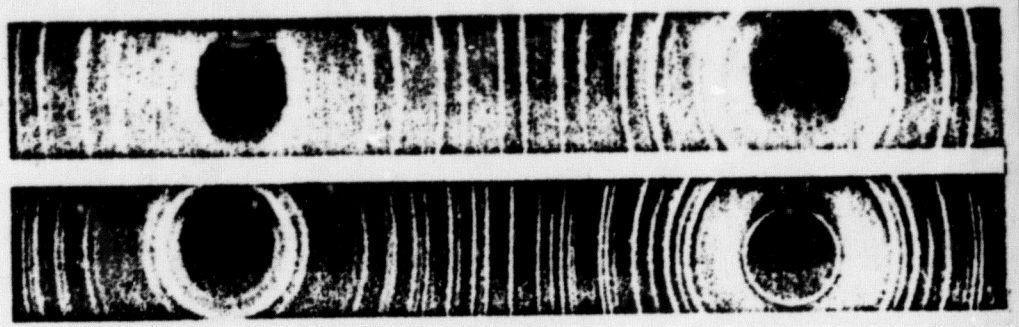
# X-RAY DIFFRACTION

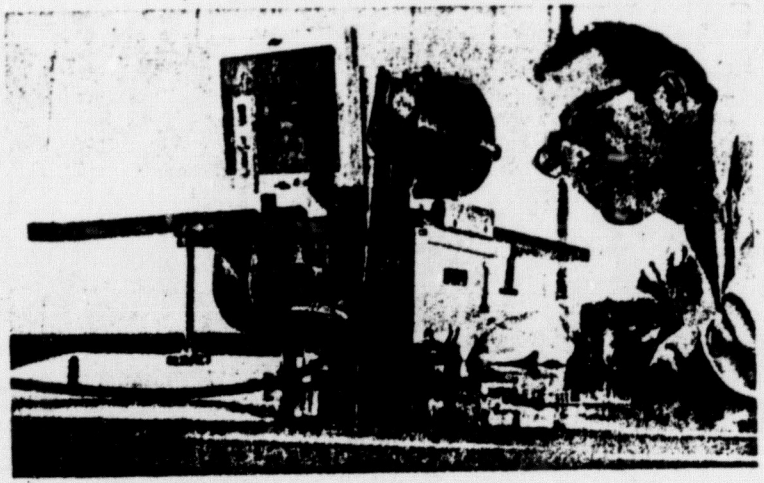
X-ray diffraction of crystalline solids serves as a non-destructive means of analyzing their basic structure.

When X-rays hit a crystalline material a spectrum is produced just as when light hits a ruled grating but the "grating" in a crystal is three-dimensional. Planes of atoms occur in all directions, depending on the configuration within the crystal. The diffraction pattern, producing spots or lines at various angles and intensities can reveal the exact position of every atom in the crystal. This enables measurement of interatomic distances and bond angles to identify the material and its anisotropic, electronic and physical properties.

Areas in which X-ray diffraction has been used in this laboratory are as follows:

- Identification of unknown materials
- Particle size line broadening
- Preferred orientation studies
- Degree of crystallinity in fibers
- Quantitative measurements of retained austenite in steels
- Microradiography
- Characterization and orientation of fibrous materials
- Orientation of metallic whiskers
- Chemical composition studies
- Determination of lattice parameters
- Quality control
- Quantitative stress measurements





E1034

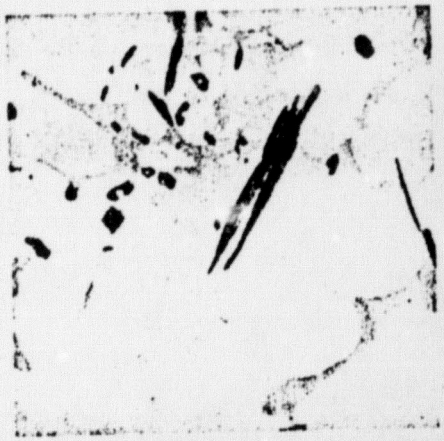
# METALLOGRAPHY-FRACTOGRAPHY-FAILURE ANALYSIS

We are fully equipped to perform metallography, fractography, and failure analysis. For specimen preparation, equipment is available for cutting, grinding, electroplating and vacuum coating, plastic mounting, mechanical or electrolytic polishing, etching, and surface replication.

The microstructure of metals, ceramics and composite materials can be examined over a wide range of magnification, by means of light microscopy, scanning electron microscopy, replicating electron microscopy, and thin film transmission electron microscopy.

Elemental analysis of secondary phases, precipitates, inclusions, segregated regions, diffusion layers, coatings, corrosion deposits and surface contaminants can be conducted by electron microprobe X-ray analysis, or by energy dispersive X-ray analysis in conjunction with the scanning electron miscroscope.

Identification of the crystalline compounds present can be made by X-ray and electron diffraction, including selected area electron diffraction. The topological features of fracture surfaces are fully revealed, and can be measured by stereoscopic light microscopy, scanning electron microscopy, and replication electron microscopy.



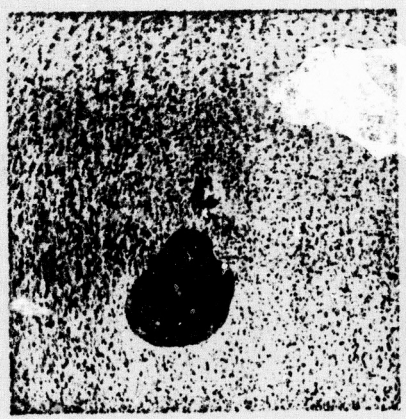
E1035

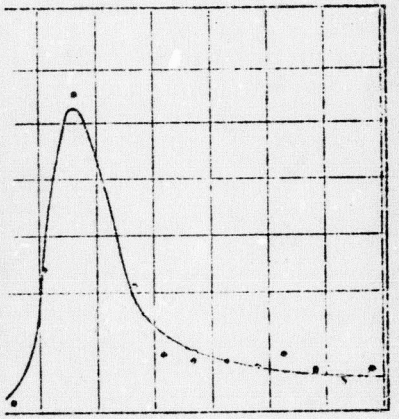
# AMONG RESEARCH APPLICATIONS

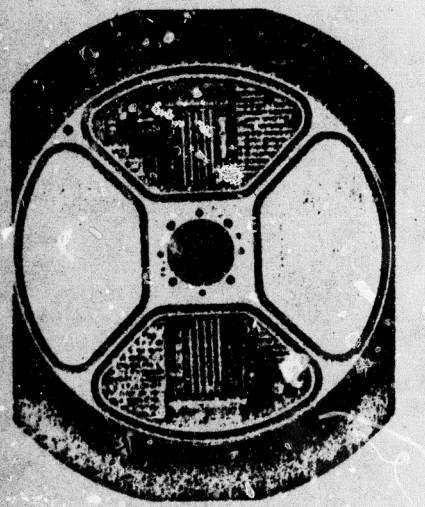
One of our specialties is high altitude particle research. We are uniquely qualified in this field because we have:

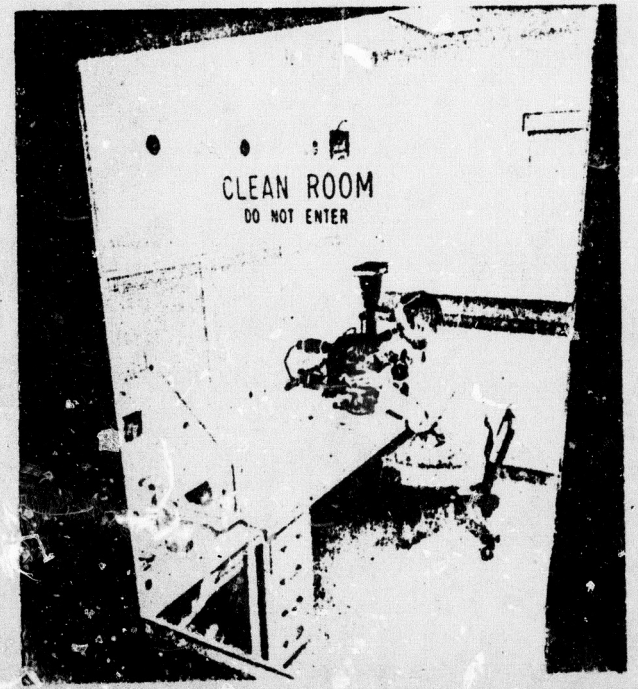
- The capabilities for building or modifying particle entrapment mechanisms.
- The techniques for preparing particle collection substrates such as super-clean metal surfaces and thin films.
- The "clean room" facilities for loading collection surfaces into flight boxes.
- The analytical equipment for small particle identification.

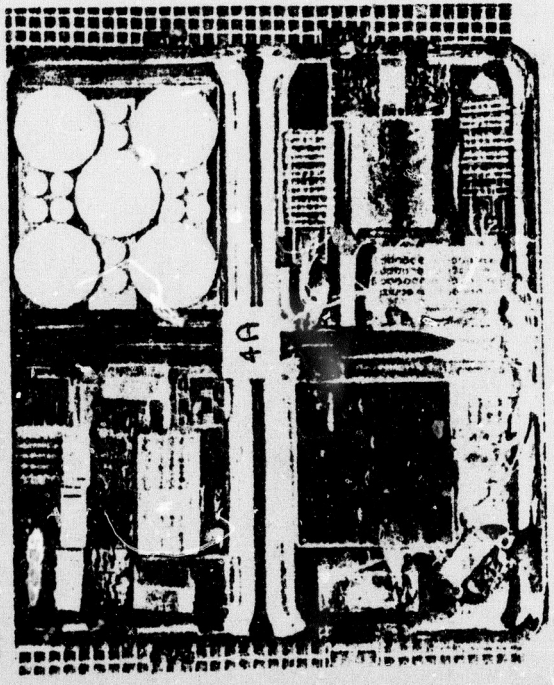
. . . and extensive experience with micrometeorite and other particle gathering projects in conjunction with flights by Aerobee, Nike, and Atlas rockets, U-2 planes, balloons, and other vehicles.









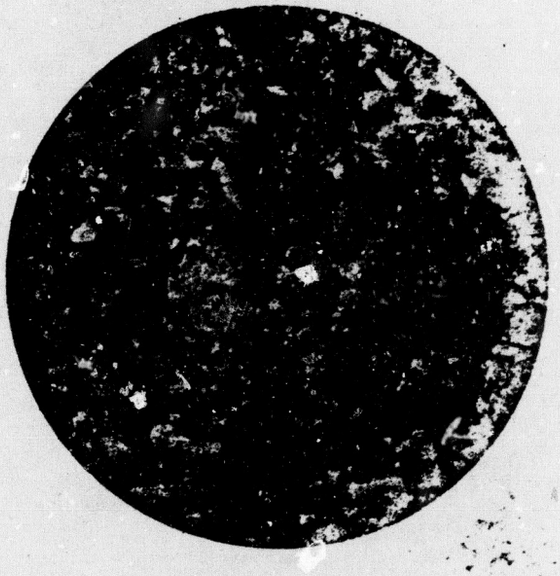
E1036

# ATMOSPHERIC POLLUTION

There are few things in the natural world whose smallest parts fail to reveal not only their unique identity, but a very special type of historical record. Even the air we breathe, burdened with the outpourings of sooty stacks and exhausts, contains tons of distinctly different particles from many sources. Under the microscope we observe, categorize and classify these particles, tracking the pollution back to its source. By microprobe analysis and electron diffraction we identify the elemental composition and chemical compounds present in specific particles. Armed with this information, enforcement agencies can take the appropriate action to remove or reduce the pollution at its source.

Typical effluent particles from polluted urban air are shown in magnifications from 300 to 10,000 diameters.

E1037



# SUPPORTI

The major services
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preparation laboratori
particle size analyzer,
centrifuge, . dessicator
tomes, micro-drill, h
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equipment, facilities
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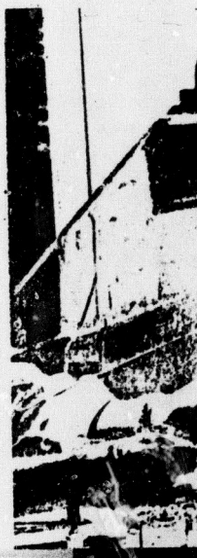

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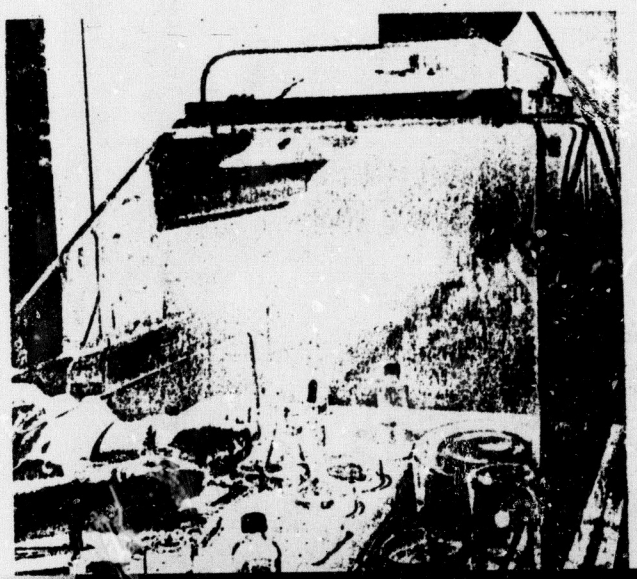

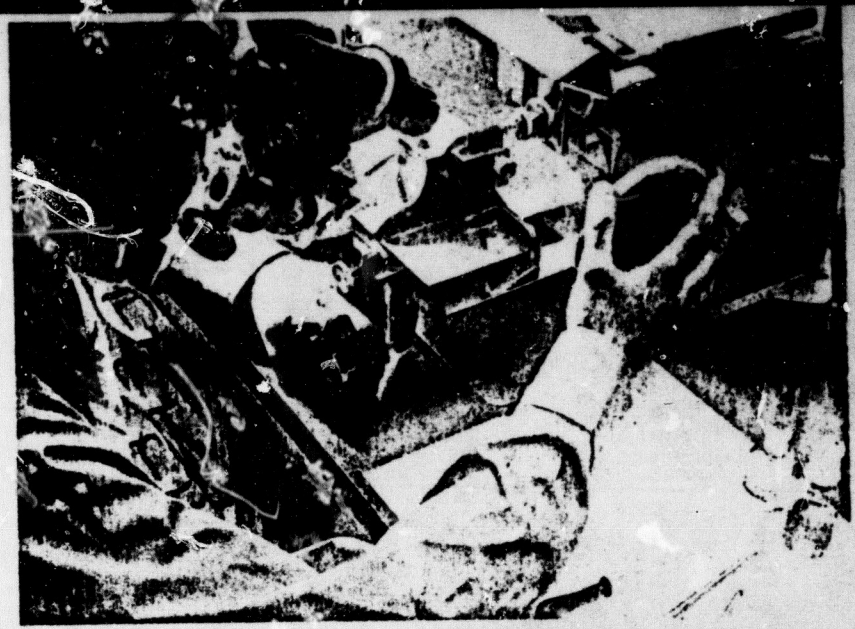

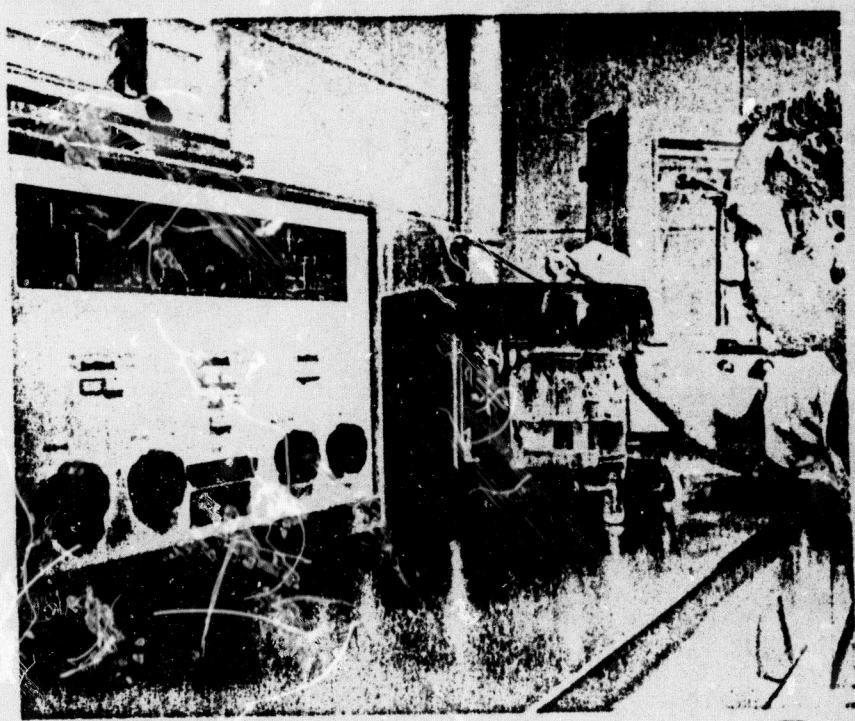

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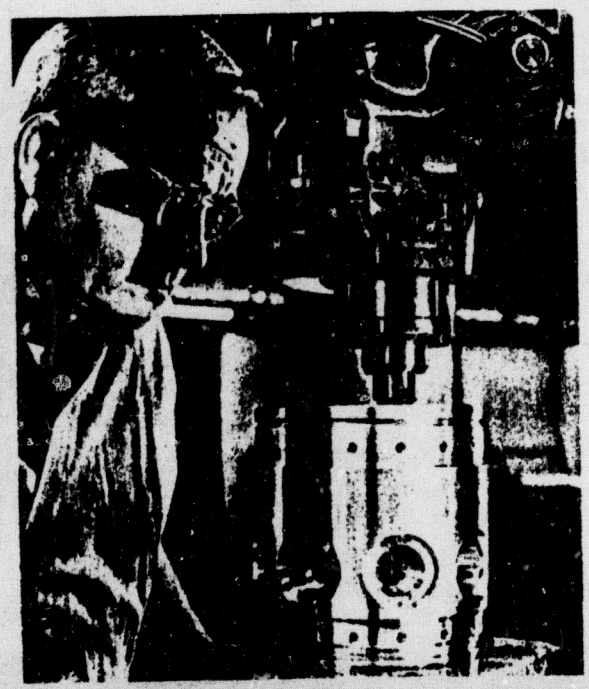

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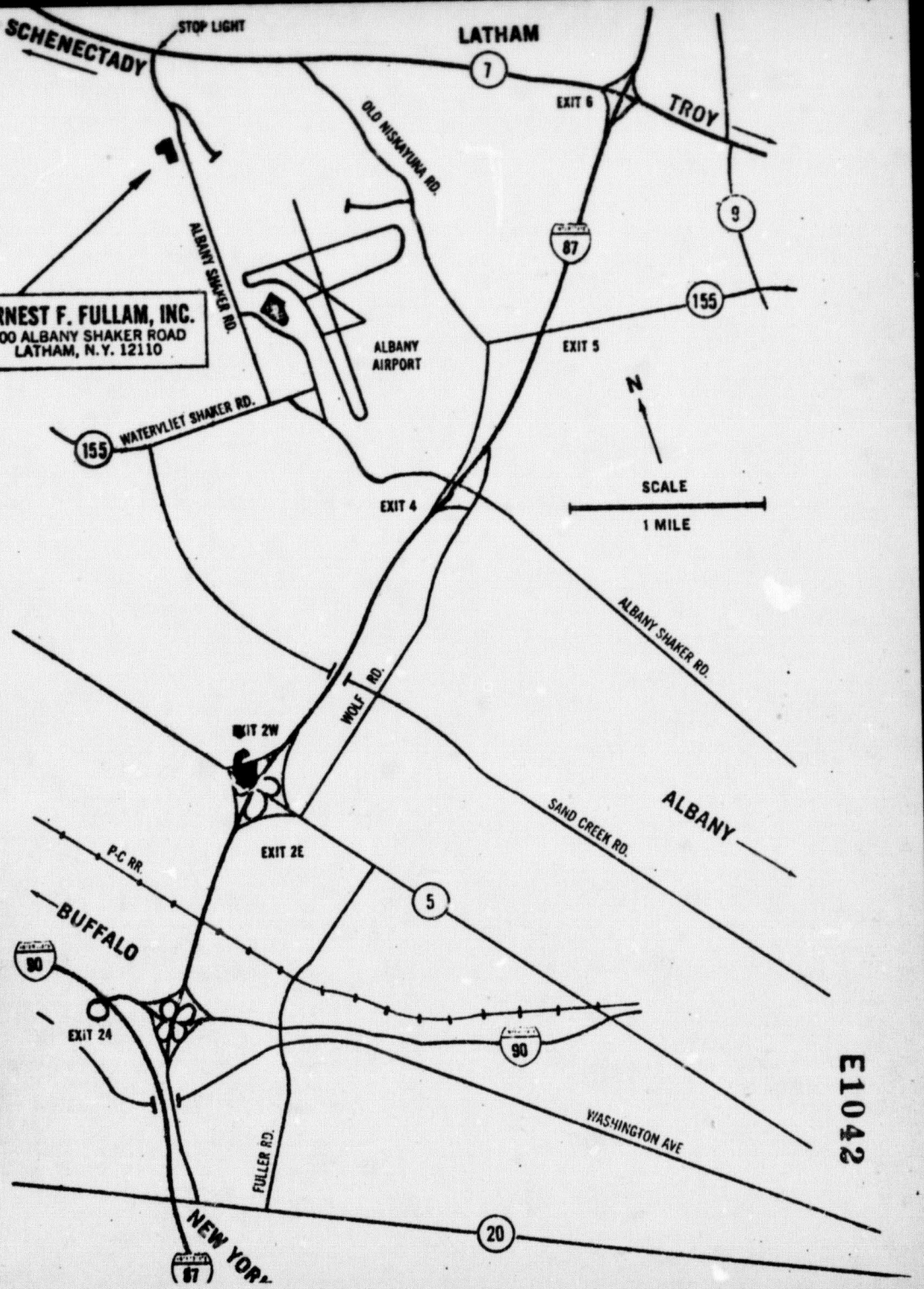


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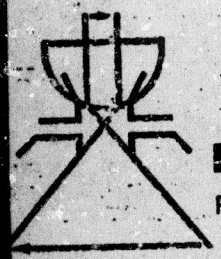

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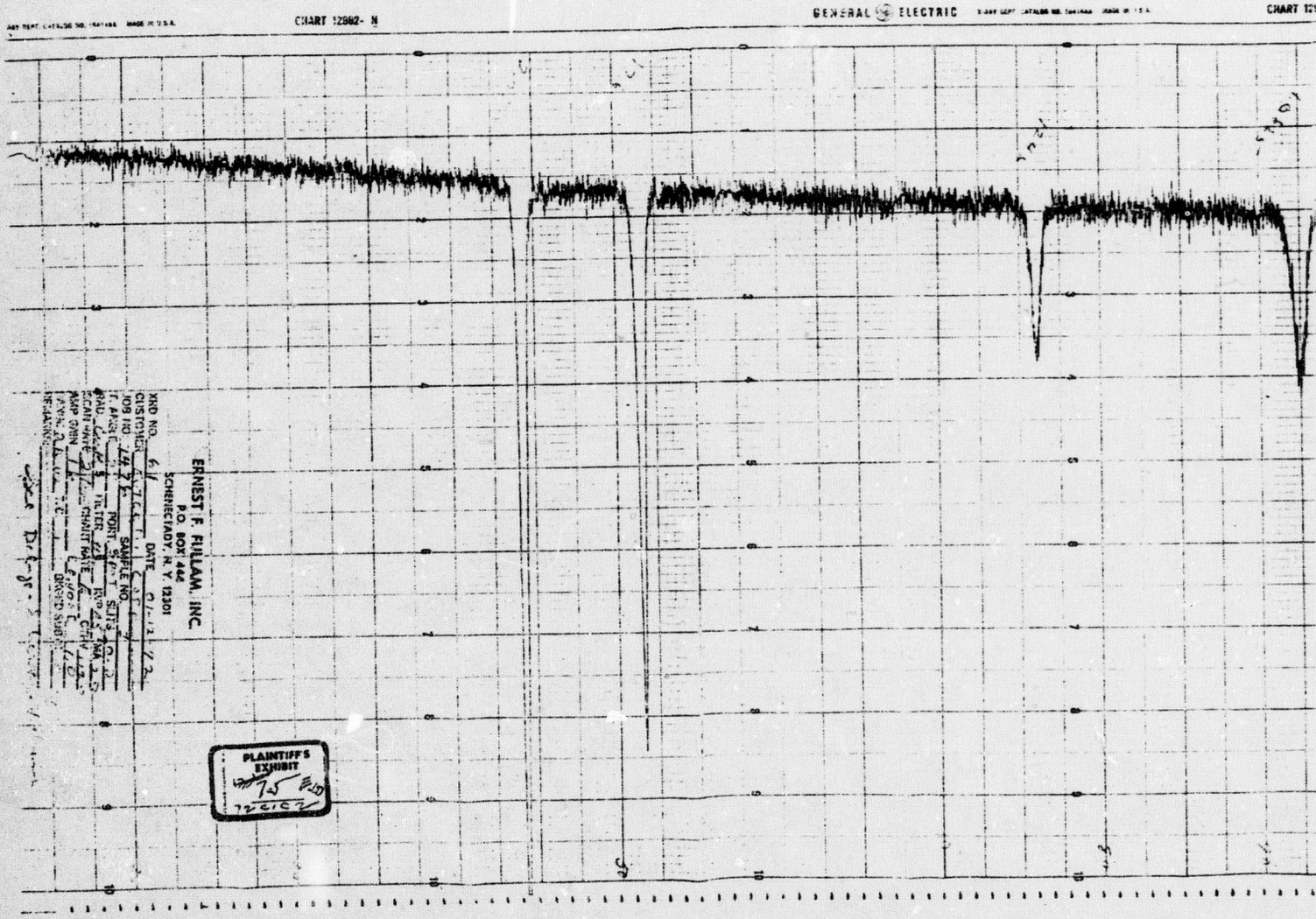
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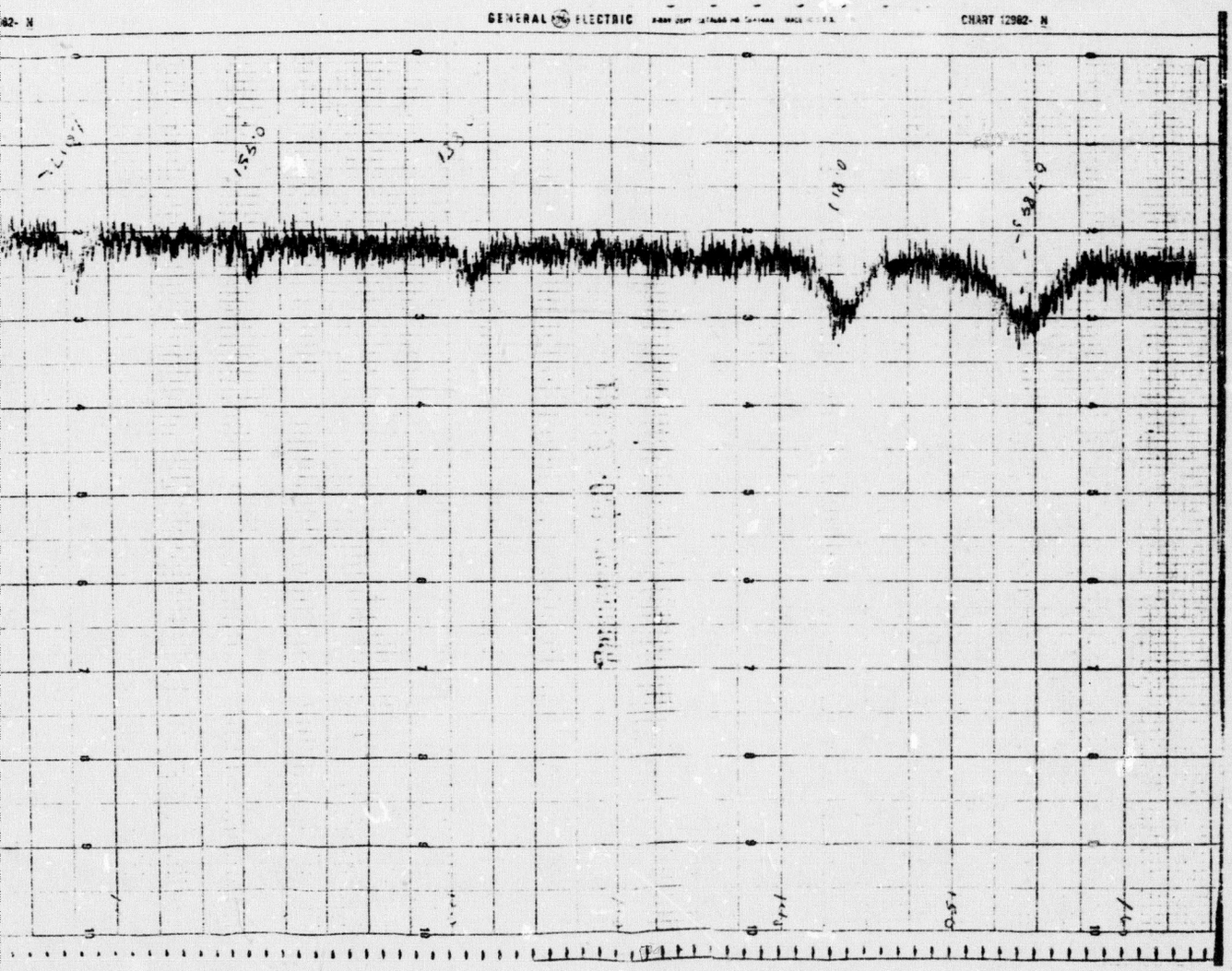

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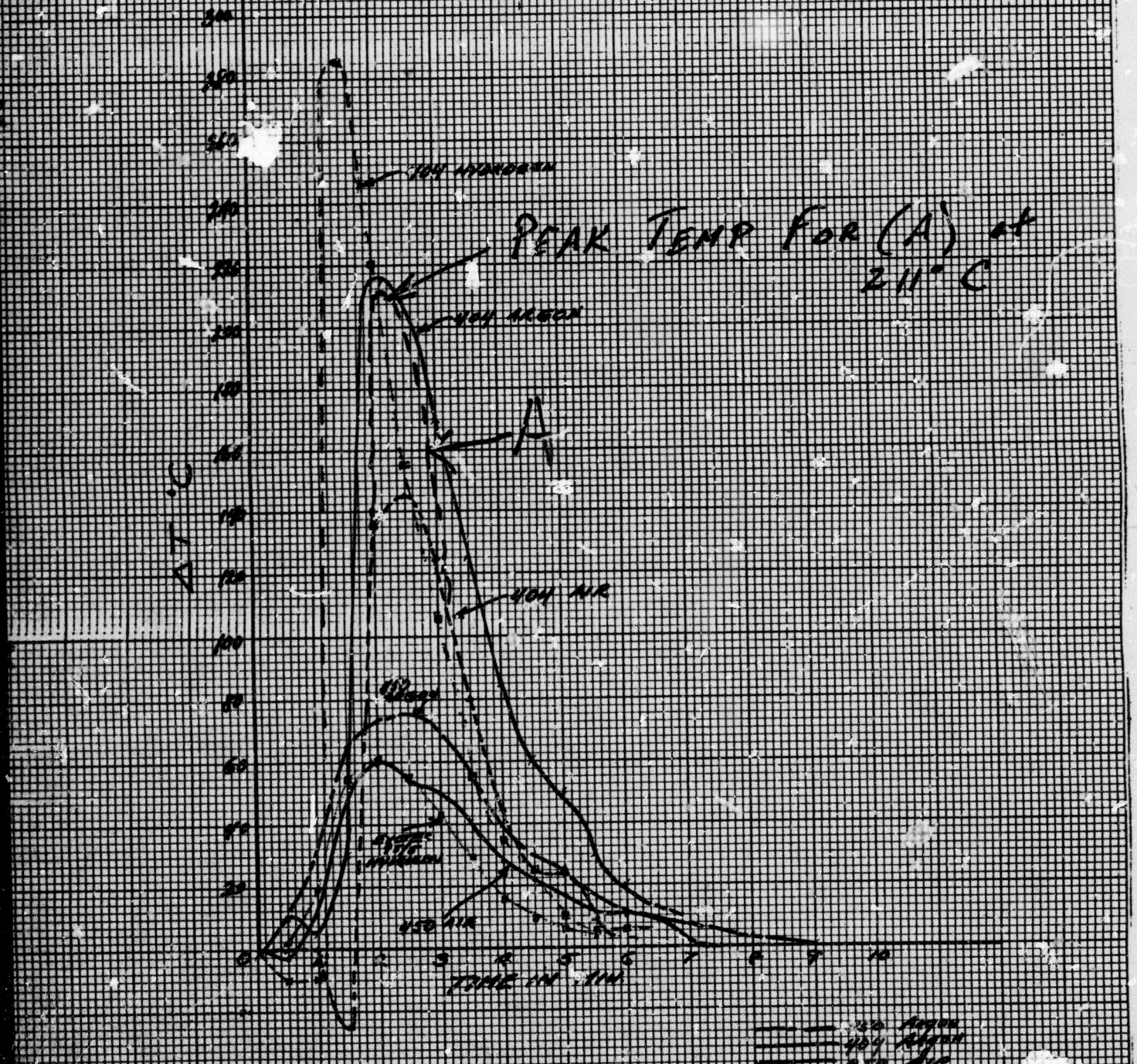

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# TYPE 5P

# ThermoSpray Gun

## Instruction Manual

METCO INC. WESTBURY, L.I., N.Y.

IN GREAT BRITAIN: METCO LTD. CHOBHAM • WOKING • ENGLAND

Instructions G 9844, Iss. b.

Cat. No. 5P 777

## SPRAYING TABLE

## METCO Type 5P ThermoSpray Gun

| Metco Powder No. | Vibrator | Nozzle Marking | Meter Valve No. | Flow Control Valve Setting (Note 1) | Spray Distance (Inches) | Lighting Pressure - PSI | | | Flow Meter Readings | | | Hourly Consumption (Note 2) | | | | Speed Sq. Ft. Covered Per Hour .001" Thick (Note 4) | Powder Weight Per Sq. Ft. .001" Thick (Note 4) | Deposit Efficiency % (Note 3) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Oxy. | Acet. | Hyd. | Oxy. | Acet. | Hyd. | Cu. Ft. Oxy. | Cu. Ft. Acet. | Cu. Ft. Hyd. | Lbs. Powder (Note 8) | | | |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 |
| 12C | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 20 | 435 | .046 | 90 |
| 14C | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 20 | 485 | .041 | 95 |
| 14E | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 20 | 485 | .041 | 95 |
| 14F | YES | P7-G | 11 | 16-18 | 5-7 | 31 | | 31 | 28 | | 56 | 54 | | 315 | 20 | 475 | .042 | 95 |
| 15C | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 20 | 490 | .041 | 95 |
| 15E | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 20 | 490 | .041 | 95 |
| 15F | YES | P7-G | 11 | 16-18 | 5-7 | 31 | | 31 | 28 | | 56 | 54 | | 315 | 20 | 475 | .042 | 95 |
| 16C | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 20 | 465 | .043 | 95 |
| 18C | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 20 | 465 | .043 | 95 |
| 31C | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 20 | 435 | .046 | 95 |
| 32C (Note 5) | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 20 | 445 | .045 | 90 |
| 34F | YES | P7-G | 11 | 16-18 | 5-7 | 31 | | 31 | 28 | | 56 | 54 | | 315 | 20 | 350 | .057 | 90 |
| 34FP | YES | P7-G | 11 | 16-18 | 5-7 | 31 | | 31 | 28 | | 56 | 54 | | 315 | 20 | 350 | .057 | 90 |
| 41C | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 14 | 360 | .039 | 95 |
| 42C | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 14 | 380 | .037 | 95 |
| 43C (Note 9) | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 18 | 440 | .041 | 90 |
| 43F (Note 9) | YES | P7-G | 11 | 16-18 | 5-7 | 31 | | 31 | 28 | | 56 | 54 | | 315 | 18 | 430 | .042 | 90 |
| 44 | NO | P7-G | 11 | 16-18 | 7-10 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 18 | 420 | .043 | 90 |
| 54 | YES | P7-G | 11 | 16-18 | 5-7 | 31 | | 31 | 28 | | 56 | 54 | | 315 | 16 | 1130 | .014 | 85 |
| 55 (Note 6) | YES | P7-G | 11 | 16-18 | 4-5 | 31 | | 31 | 28 | | 56 | 54 | | 315 | 16 | 380 | .042 | 95 |
| 56F (Note 9) | YES | P7-G | 11 | 16-18 | 5-7 | 31 | | 31 | 28 | | 56 | 54 | | 315 | 20 | 490 | .041 | 95 |
| 101 (Note 9) | YES | P7-G | 12 | 6-10 | 2-3 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 3 | 165 | .018 | 90 |
| 105 (Note 9) | YES | P7-G | 12 | 6-10 | 2-3 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 2½ | 130 | .019 | 80 |
| 201 (Note 9) | YES | P7-G | 12 | 6-10 | 2-3 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 2½ | 110 | .023 | 80 |
| 210 | YES | P7-G | 12 | 6-10 | 2-3 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 3 | 80 | .038 | 90 |
| 211 | YES | P7-G | 12 | 6-10 | 2-3 | 20 | 13 | | 34 | 34 | | 60 | 33 | | 5 | 125 | .039 | 95 |
| 404 (Notes 7 & 9) | YES | P7-B | 12 | 16-18 | 7-10 | 17 | 12 | | 32 | 32 | | 48 | 33 | | 10 | 230 | .043 | 75 |
| 410 | YES | P7-D | 12 | 14-16 | 3-5 | 17 | 12 | | 32 | 32 | | 48 | 33 | | 4 | 145 | .027 | 70 |
| 411 | YES | P7-D | 12 | 14-16 | 3-5 | 17 | 12 | | 32 | 32 | | 48 | 33 | | 4 | 115 | .035 | 70 |
| 412 | YES | P7-D | 12 | 14-16 | 3-4 | 17 | 12 | | 32 | 32 | | 48 | 33 | | 4 | 95 | .043 | 60 |
| 413 | YES | P7-D | 12 | 14-16 | 3-4 | 17 | 12 | | 32 | 32 | | 48 | 33 | | 4 | 90 | .045 | 60 |

Hottest

*NOTES:*

1. The ranges of Flow Control Valve settings are nominal. Individual guns may require settings which fall outside the range. Adjust as necessary to obtain the best coatings.
2. The consumptions shown are optimum values obtained by skilled operators with all equipment in first-class condition.
3. The deposit efficiency figures shown are approximate values which have been rounded off for easy calculation. Where accurate values are important, special on-the-job test runs are advised.
4. The figures shown in columns 17 and 18 for 12C, 14C, 14E, 14F, 15C, 15E, 15F, 16C, 18C, 31C, 34F and 34FP are based on fused coatings.
5. The figures for 32C are based upon a coating in the as-sprayed condition. To fuse this material, an overcoat of 15E must be applied. Refer to the Flame Spray Handbook for further information.
6. Cooling air is required with No. 55 Powder. Orient the air cap with cooling end (flat face) outward.
7. With 404 and 404-NS Powders, proper spray distance is especially important. Bond strength and reliability fall off rapidly outside of the recommended range.
8. For convenience, metal powder spray rates can be reduced to approximately half rate by replacing the No. 11 Metering Valve with the No. 12 Metering Valve. Lighting pressures and flow readings remain unchanged.
9. N.S. grade powder uses the same spray parameters as powder of the same number without the N.S. suffix.

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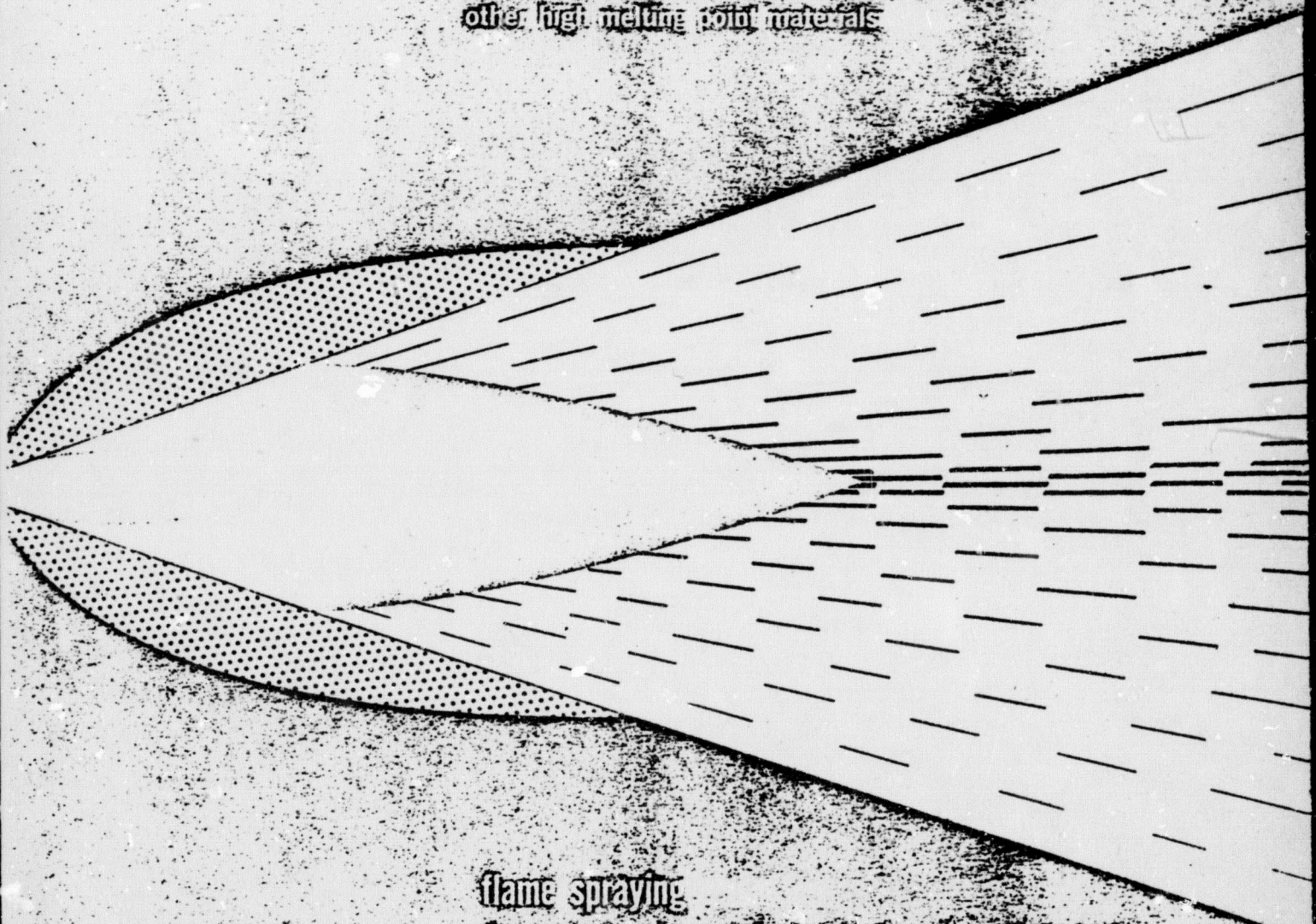
# THE METCO FLAME SPRAYING PROCESSES

Basic engineering and application data on flame sprayed coatings of metals, ceramics, carbides and other high melting point materials



## flame spraying

this term has been adopted to describe any process whereby a material is brought to its melting point and sprayed onto a surface to produce a coating. Three processes now include:

1 Metallizing 2 ThermoSpray 3 Plasma Flame

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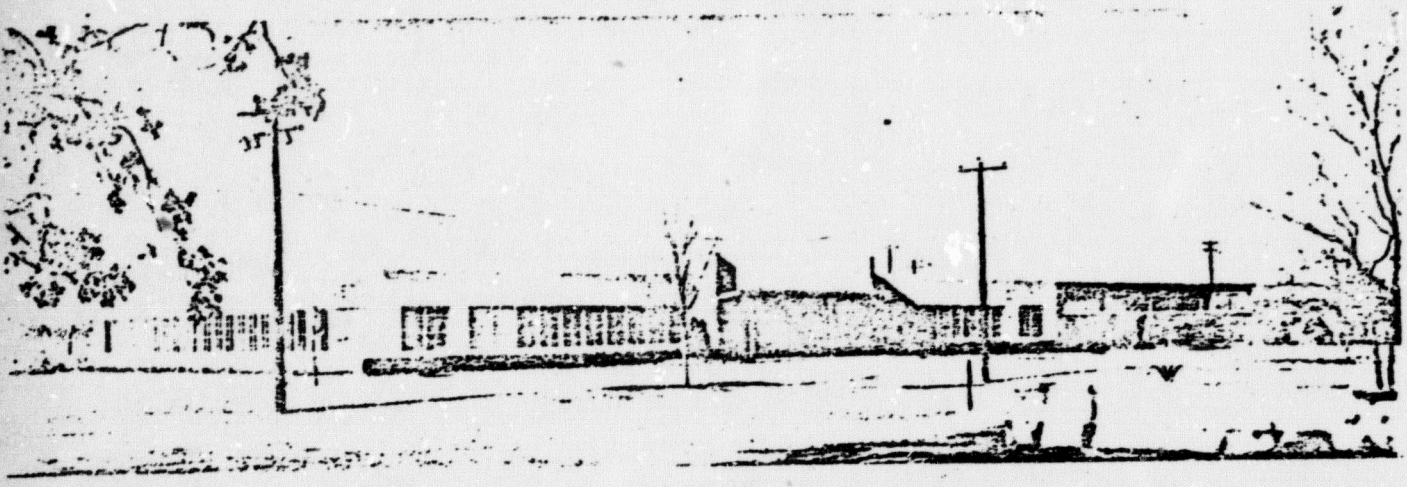

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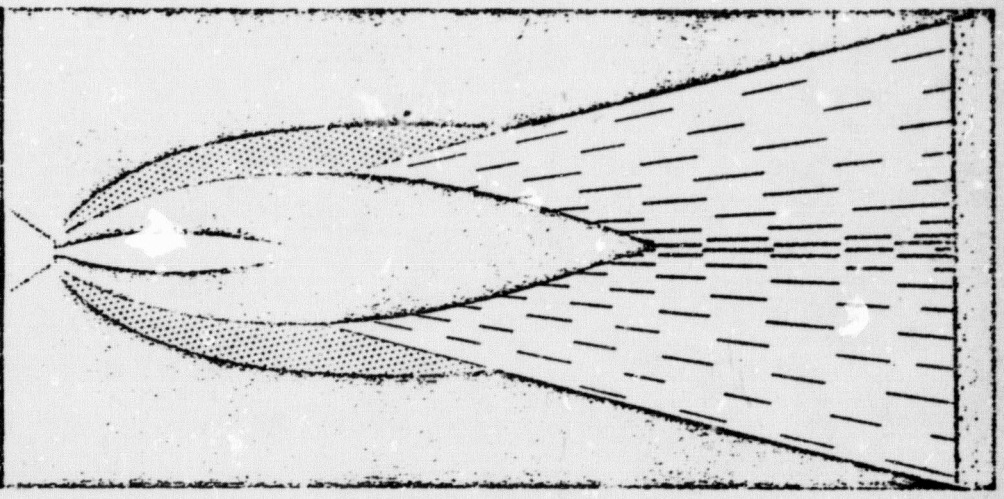


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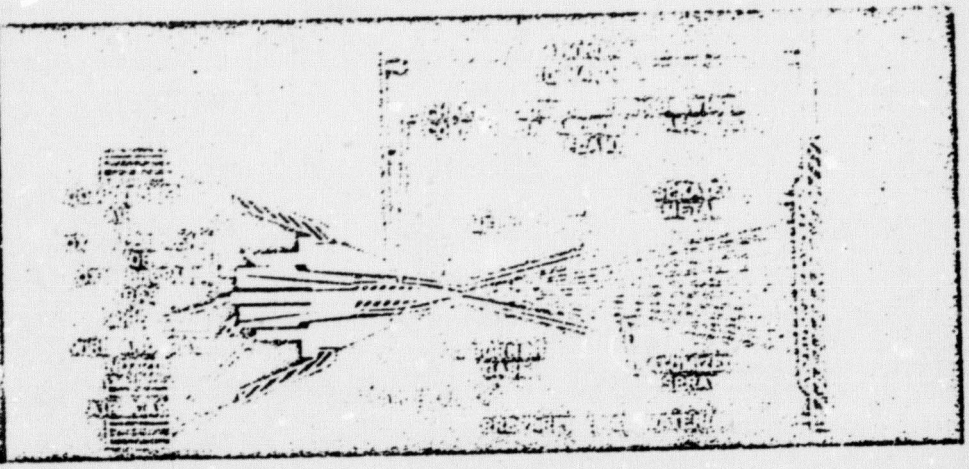
# THE METALLIZING PROCESS— HOW IT WORKS



Wire nozzle and air cap cross section, METCO Wire Metallizing Gun

The term "metallizing" is used to describe that type of flame-spraying process which involves the use of metal in wire form. The wire is drawn through the gun and nozzle by a pair of powered feed rolls. Here the wire is continually melted in the oxygen-fuel-gas flame and atomized by a compressed air blast which carries the metal particles to the previously prepared surface. The individual particles mesh to produce a coating of the desired metal. This meshing action is still not completely understood, but the effect is apparently due to a combination of mechanical interlocking and cementation of the oxides formed during the passage of the particles from the gun nozzle to the sprayed surface.

Any metal that can be drawn into wire form can be sprayed with the Metco Metallizing Guns.

A list of typical metals available for wire metallizing is carried in Table 5—Spraying Data. For information on the flame-spraying of metals and other materials that cannot be drawn into wire see Metco ThermoSpray and Plasma-Flame Processes on pages 7 and 11 respectively.

Metallized coatings have been in extensive use for over a quarter of a century. The process was originally used primarily for repair and maintenance of machine parts. However, their desirable characteristics exhibited under severe service conditions has led a rapidly growing number of product designers to take advantage of the special properties of these coatings. They are used to improve wear resistance, abrasion resistance, corrosion resistance and for weight reduction as well as lowered production costs. In addition, the economic feasibility of applying comparatively expensive metals in thin coatings at critical areas is well known. This provides the design engineer with an economical means of imparting a wide range of desired mechanical and physical characteristics to particular parts of a product, and materially increases flexibility of design.

Result: Many companies are using this unique process in the manufacture of original equipment. Many others have assigned their research and development departments to investigate its profit potentials in product improvement, increased production rates, lower manufacturing costs.

The various phases of metal spray can be very accurately controlled when using modern metallizing guns and auxiliary equipment. Results can be duplicated exactly since all factors that affect the spraying are under precise control. The supply and pressure of oxygen-fuel gas, and air are accurately controlled by flowmeters and pressure regulators. Wire feed is closely adjustable and can be maintained at a predetermined rate of feed.

Specific characteristics of metallized materials are treated in the tables that follow. However it should be noted that for mechanical applications the composition of sprayed metal is chemically and physically different from that of the original wire. In general it is harder and more porous than its equivalent in cast or wrought form. This combination of hardness and microscopic surface porosity which tends to hold lubricants, makes the metallized coating an ideal bear-

ing surface, much superior to the same metal in its original form.

## coating thickness

Theoretically there is no limit to the thickness of the metallized coating which may be applied. However, in practical applications internal stresses set up in metallized coatings, some of which show shrinkage characteristics that tend to raise the edges of the coating on flat surfaces where unusual thicknesses have been sprayed over large areas. This limitation again is extremely variable and it is recommended that wherever possible, special provisions for edge bonding be worked out experimentally. (For further details see Metco Handbook). It should be noted that these considerations do not apply on cylindrical shapes where metallized coatings of any reasonable thicknesses have been applied.

## apparent hardness versus true hardness and abrasion resistance of metallized coatings

Readings obtained with hardness testing equipment usually available are only useful in comparing one sprayed metal with another just as with other materials, such as sintered powder metal. For instance, Table One gives the Rockwell Hardness of a number of sprayed metals, and though 0.80 carbon steel is very hard, when sprayed it develops a Rockwell reading of only C-36. Actually the true particle hardness is C-67 and the metallized coating will resist wear much better than hardened steel because of the surface porosity which holds the lubricant.

## typical applications

### wear resistance

A typical example of the superior wear resistance offered by metallized coatings of superior metals is offered in the example of a manufacturer of precision bearings. The extremely close tolerances specified for the final product prohibited the use of ball-bearing equipped grinder-spindles. Consequently split-sleeve bearings were employed, and these were kept so tight that ordinary uncoated spindles had to be replaced because of wear within a few days to less than a month. Many different types of special steels were tried without success. Now the spindles are metallized with Sprabond (molybdenum) which is sprayed directly on the spindle surface. These metallized spindles commonly are in service for over a year and a half before they are removed and re-metallized.

## TABLE 1—HARDNESS

| Metal | Rockwell Hardness |
|---|---|
| SPRABRONZE AA | B 78 |
| SPRABRONZE TM | B 50 |
| METCO COPPER | B 32 |
| METCOLOY #1 (18-8 Stainless) | B 78 |
| METCOLOY #2 (High Chrome Stainless) | C 29 |
| SPRASTEEL LS (Low Shrink) | C 25 |
| SPRASTEEL #10 (0.10 Carbon) | B 89 |
| SPRASTEEL #25 (0.25 Carbon) | B 90 |
| SPRASTEEL #80 (0.80 Carbon) | C 36 |
| METCO NICKEL | B 49 |
| METCO MONEL | B 39 |
| SPRABOND (Molybdenum) | C 38 |

## TABLE 2—TENSILE STRENGTH

| Metal | Ult. Str. psi | Strain at U.T. % Increase in Length |
|---|---|---|
| METCO ALUMINUM | 19,500 | 0.23 |
| METCO S.F. ALUMINUM (Super Finish) | 37,000 | 0.54 |
| SPRABRONZE AA (Al. Bronze) | 29,000 | 0.46 |
| SPRABRONZE TM | 26,500 | 0.56 |
| METCOLOY #1 (18-8 Stainless) | 30,000 | 0.27 |
| METCOLOY #2 (High Car. Stainless) | 40,000 | 0.50 |
| SPRASTEEL LS (Low Shrink) | 33,000 | 0.54 |
| SPRASTEEL #10 (0.10C) | 30,000 | 0.30 |
| SPRASTEEL #25 (0.25C) | 34,700 | 0.46 |
| SPRASTEEL #80 (0.80C) | 27,500 | 0.42 |
| SPRABOND (Molybdenum) | 7,500 | 0.30 |

## TABLE 3—COMPARATIVE SHRINK

| Metal | Shrink (in./in.) |
|---|---|
| SPRABRONZE AA (Al. Bronze) | 0.005 |
| SPRABRONZE TM | 0.009 |
| METCOLOY #1 (18-8 Stainless) | 0.012 |
| METCOLOY #2 (High Carb. Stainless) | 0.002 |
| SPRASTEEL LS (Low Shrink) | 0.002 |
| SPRASTEEL #10 (0.10C) | 0.008 |
| SPRASTEEL #25 (0.25C) | 0.006 |
| SPRASTEEL #80 (0.80C) | 0.002 |
| SPRABOND (Molybdenum) | 0.003 |

## TABLE 4—BOND STRENGTH

| Metal Sprayed | Type Test | Grit Blast | Metco Method | Sprabond over Smooth Surface | Sprabond over 24 Pitch Thread |
|---|---|---|---|---|---|
| Sprasteel #10 (0.10C) | Shear | 6970 | 14600 | 8850 | 16000 |
| | Tension | 2661 | 4580 | 2150 | 3050 |
| Sprabronze TM | Shear | 4890 | 6930 | 5460 | 6950 |
| | Tension | 1150 | 2120 | 1820 | 2150 |
| Metcoloy #1 (18-8 Stainless) | Shear | 8322 | 18913 | 9500 | 20500 |
| | Tension | 3040 | 6062 | 2200 | 3050 |
| Metco Aluminum | Shear | 2830 | 4500 | 4400 | 5500 |
| | Tension | 1032 | 2142 | 1640 | 2400 |
| S.F. Aluminum (Super Finish) | Shear | 4500 | 7870 | 5100 | 8150 |
| | Tension | 1708 | 2920 | 2050 | 3100 |

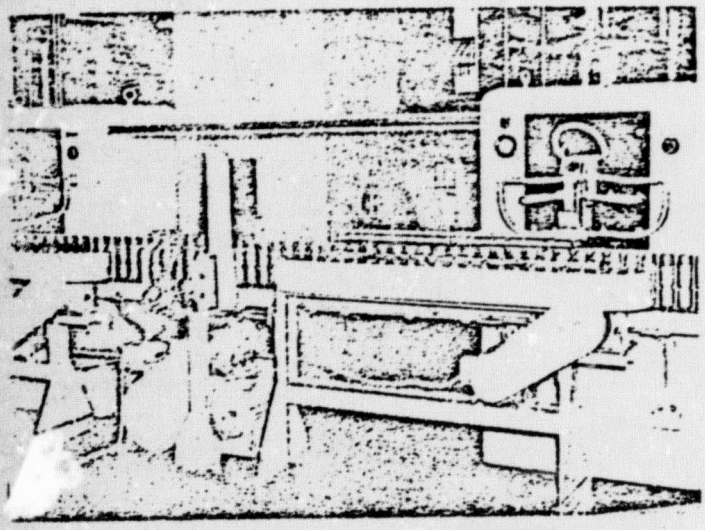
Note: Figures above show stress in psi required to break bond of various types of metals and surface preparations. Base specimens were cold finished steel. Non-metallic bases cannot be tested because, with proper preparation, they will fracture before bond failure occurs.

A large glass company manufactures the heating elements for a radiant heater. The heating element consists of a sheet of glass onto which is sprayed a continuous strip of aluminum in the form of a flat coil.

Another glass company has been metallizing the edges of meter glasses for many years. The object here is to enable the glass to be soldered to the metal casing so as to provide a joint that is tamper-proof, gas-proof and moisture-proof. A simple production set-up enables one gun to produce some 6,000 pieces a day. Metal used is ordinary brass or copper.

The above are representative of the activities of some of the large companies. There are others who are using metallizing for less-known products such as road signs, catapnotes and similar items.

lower costs—higher production



These automotive valves are being metallized with aluminum to provide superior heat protection. In the assembly line pictured here they are automatically conveyed past pre-heating torches and rotated past metallizing machines spraying pure aluminum. This method has substantially increased production rates and lowered manufacturing costs over the previously used dip-tank method.

## miscellaneous production applications

### brazing and soldering

Metallizing is often a very satisfactory way of pre-placing solder or brazing metal. Lead solders, silver solder, aluminum brazing metals and pure copper can be applied in this manner. The usual practice is to spray the filler metal in place using ordinary metallizing techniques. The assembly is generally furnace-brazed using a hydrogen furnace.

### coatings on non-metallic materials

A number of products are on the market which incorporate metallized glass. A nationally advertised double window glass which consists of sheets of plate glass soldered together with an air space between is metallized. A copper alloy is sprayed in a 1/4" band along the edges—two sheets are placed together with a metal spacer in between. The spacer is in contact with the metallized copper edges and then a special solder is flowed on to effect a complete seal.

## miscellaneous non-metallic materials

### carbon brushes and resistors

Carbon resistors and brushes are sprayed with a thin film of copper as a means of obtaining an electrical connection of high conductivity. Pigtails are then soldered to this sprayed copper surface.

### ceramic resistors and insulators are also sprayed for the same reason

Most of this work is done on a high production basis in automatic machines. No preparation is needed since the copper bonds directly to the relatively porous surface of the unglazed ceramic.

### cloth and paper

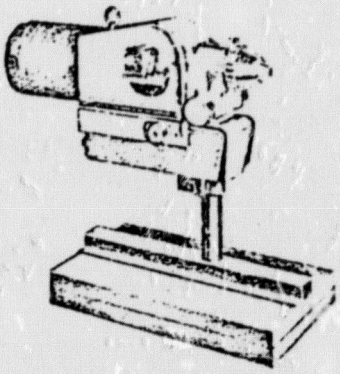
Cloth and paper are metallized with the lower melting point metals such as aluminum and zinc for a number of different uses, including electrolytic condenser plates where aluminum is sprayed on both sides of a gauze-like fabric. No preparation is required since the metal inter-locks with the base materials.

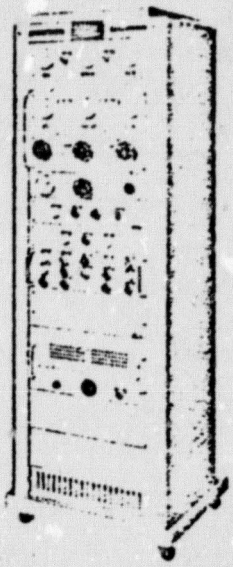
## metallizing equipment

METCO Metallizing Guns are available in three types—one, an all-purpose metallizing gun designed for tool-post mounting or hand-held operation; another especially designed for high-speed hand spraying in corrosion protection work; and the Type K Electric Drive Metallizing Machine, designed for production metallizing where heavy duty, continuous operation is required.

E1055

# FULLY AUTOMATIC PRODUCTION METALLIZING





**METCO TYPE K heavy duty metallizing machine and TYPE C Automatic Control System**

New electronic controls such as the METCO Type C Control Systems, have made fully automatic push-button metallizing a reality. These electronic units not only start and stop the metallizing guns, change the flame to "idle" when desired, and control and start the wire feed, but they literally watch and supervise the operation of the metallizing gun.

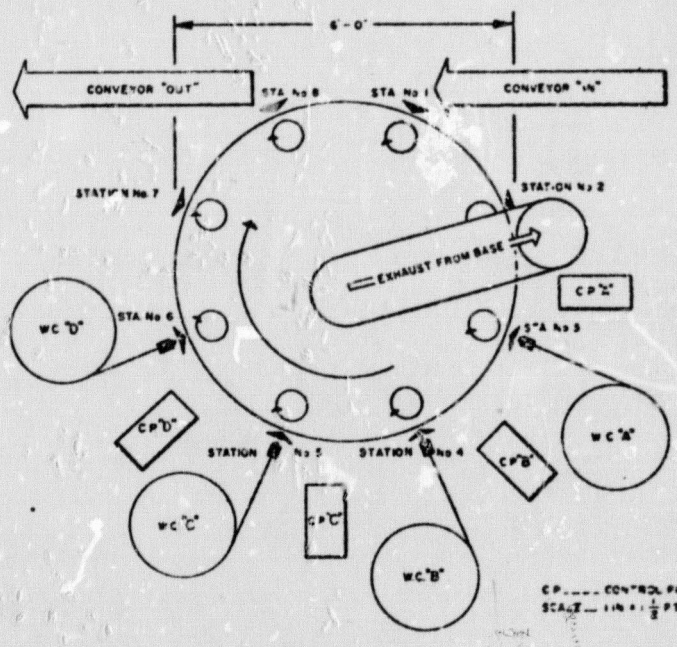
One control panel is used for each metallizing gun, and any number of guns and panels can be installed and inter-locked, so that all of the units can be controlled from a single master station.

## typical automatic set-up

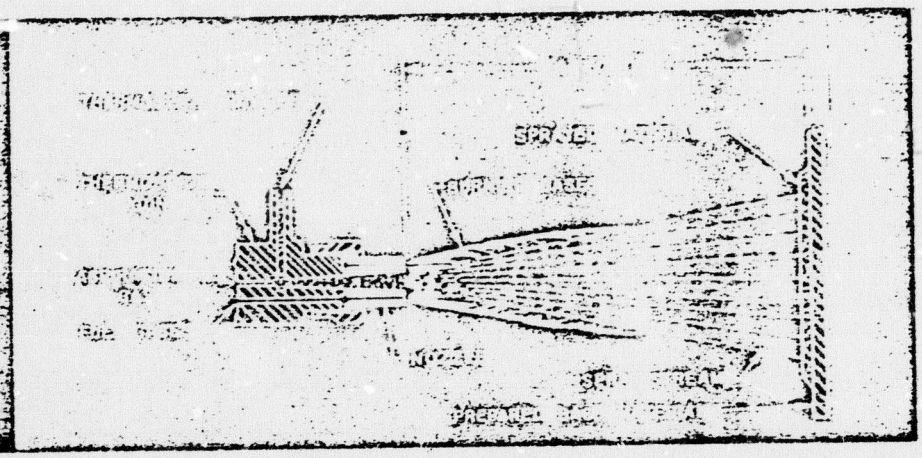
This installation was developed to apply a 3/4" wide bronze bearing surface to the center of a shaft 4" in diameter and 18" long. Coating thickness is 1/8". Pieces are undercut 1/32", build-up is 1/8" and approximateley 1/32" removed in finishing, providing a band approximately 1/16" larger than the shaft diameter. Eight stations are provided and the following operation takes place at each:

| | |
|---|---|
| Station No. 1 | loading |
| Station No. 2 | preheat to approximately 250° F. |
| Station No. 3 | apply SPR. BOND .0015" to .002" thick. Spray time 15 to 20 seconds |
| Station No. 4 | SPRABRONZE AA, first coating |
| Station No. 5 | SPRABRONZE AA, second coating |
| Station No. 6 | SPRABRONZE AA, third coating |
| Station No. 7 | inspection, or spare for increased production |
| Station No. 8 | unloading |

This machine indexes once every 60 seconds and 10 seconds are allowed for this operation. An automatic timing device shuts down the wire feed at B, C, and D for this intermediate period. Metallizing Machine A operates for 15 to 20 seconds and the wire feed is shut off for 40 to 45 seconds.



# THE THERMOSPRAY PROCESS— HOW IT WORKS



## METCO ThermoSpray Powder Gun

The term "ThermoSpray" is used to describe that type of flame-spraying equipment which involves the application of metals, and other materials in powder form. These include alloys of nickel-chromium-boron-silicon, tungsten-carbide mixtures, ceramics such as alumina and zirconia, and certain cermets.

The powdered materials are held in a hopper atop the gun and gravity fed into the gun proper where they are picked up by the oxy-acetylene (or hydrogen) gas mixtures and carried to the gun nozzle. Here they are melted almost instantly due to the extremely high thermal efficiency of the gun and carried to the surface being sprayed by means of a siphon-jet arrangement at the gun nozzle. This also contributes to its thermal efficiency. Very high deposit efficiencies can be attained, (usually well above 90%). The mechanical design of the gun is simplicity itself for extremely long life with minimum maintenance requirements.

## materials that can be sprayed by the ThermoSpray process

There are three basic types of coating materials which are used in the various ThermoSpray processes. Although there are many possible combinations, and some overlap between types, these are:

1. Alloys for fused coatings
2. Oxidation-resistant metals and alloys, used in either an "as sprayed" or fused state.
3. Ceramics.

The following descriptions cover those materials most commonly used in the ThermoSpray processes. This list is subject to expansion and many special purpose powders, which are not described, are presently available.

### Alloys for fused coatings

Alloys in this group are called "self-fluxing." Basically they are chromium-nickel-alloys to which boron and silicon fluxing agents have been added. These affect fluidity and surface tension of the molten alloys during fusing operations, an important consideration because in the past, alloys in this class have been characterized by rapid transition from solid to liquid —in other words, a narrow "plastic range"—making them difficult to fuse without sagging. Metco has modified several alloys to provide a wide plastic range and enable the user to fuse heavy coatings without difficulty.

These alloys range in hardness from Rc30 to Rc65. They are highly resistant to wear and abrasion and will often outwear steel of equivalent hardness by as much as 20 to 1. They are also highly resistant to corrosion and oxidation, in this respect, being generally superior to premium stainless steels.

### Tungsten carbide

ThermoSpray Powders 32C and 31C consist of high-quality sintered tungsten carbide blended with a self-fluxing alloy. 32C provides coatings having the maximum carbide content obtainable in coatings of this type, while 31C is blended with a higher proportion of the self-fluxing matrix material for use where maximum carbide concentration is not required. In these coatings, as fused, the carbide grains are securely bonded in a matrix material which, itself, is outstanding in wear resistance. Careful classifica-

tions of selected grades of carbide produces very high deposit efficiencies. Hardness of the matrix in such coatings is RC 62-66 while the carbide is the equivalent of RC 75.

These powders are recommended for application where abrasion conditions are severe.

### Oxidation resistant alloys

These alloys were developed primarily on the basis of their oxidation resistance and then processed to meet the requirements of the ThermoSpray process. They are not self-fluxing and are always used in the "as-sprayed" state. In many cases they are used as bonding or barrier coatings where a ceramic will subsequently be applied.

### Ceramics

The high thermal efficiency of the ThermoSpray gun makes it possible to apply refractory materials at speeds which are economically feasible. Many special purpose refractory and cermet coatings may be sprayed, but alumina and zirconia are the materials most widely used.

ThermoSpray Powder 101, a grey alumina containing titania and traces of other oxides which tend to "toughen" the grain. This material is used for wear-resistant coatings and for heat resistance where wear and abrasion are also present.

ThermoSpray Powder 105 – a high purity white alumina having lower emissivity and lower thermal conductivity than No. 101. Preferred for its thermal properties in heat barriers, particularly where blast erosion is a factor. Also used as a high temperature dielectric although sealing is required if dielectric strength is to remain constant.

ThermoSpray Powder 201 — lime-stabilized zirconia used for heat insulation. Melting point approx. 4650 F. Color — light yellow. Abrasion resistance fair, though lower than that of ThermoSpray 101 or 105.

**TYPE ANALYSIS OF METCO FLAME SPRAY POWDERS**

| METCO Metallic Powders | Tungsten Carbide Cobalt Aggregate | Iron | Nickel | Chromium | Carbon | Silicon | Boron | Copper | Molybdenum | Aluminum | Cobalt | Chromium Carbide |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| For Fused Coatings | | | | | | | | | | | | |
| 12C | – | 2.5 | Bal | 10. | .15 | 2.5 | 2.5 | – | – | – | – | – |
| 14E–14F | – | 4. | Bal | 14. | .6 | 3.5 | 2.75 | – | – | – | – | – |
| 15E*-15F* | – | 4. | Bal | 17. | 1. | 4. | 3.5 | – | – | – | – | – |
| 15C*†† | – | 2.5 | Bal | 16. | .5 | 4. | 4 | 3. | 3. | – | – | – |
| 18C | – | 2.5 | Bal | 18 | .2 | 3.5 | 3. | – | 6. | – | 40. | – |
| 31C | 35. | 2.5 | 45 | 11. | .5 | 2.5 | 2.5 | – | – | – | – | – |
| 32C | 80. | .8 | 14. | 3.5 | .1 | .8 | .8 | – | – | – | – | – |
| 34F-34FP | 50. | 3.5 | 33. | 9. | .5 | 2. | 2. | – | – | – | – | – |
| METCO Metallic Self-Bonding Powders | | | | | | | | | | | | |
| 404* | – | – | 80. | – | – | – | – | – | – | 20 | – | – |
| 439* | 50. | 1.5 | Bal | 6. | .5 | 1.5 | 1. | – | – | 3 | – | – |
| 450† | – | – | Bal | – | – | – | – | – | – | 4.5 | – | – |
| 451† | – | – | Bal | 9.5 | – | 2.5 | 1.5 | – | – | 0.5 | – | – |
| METCO Metals, Alloys and Mixtures generally used "as Sprayed" | | | | | | | | | | | | |
| 41C | – | Bal | 12. | 17. | .1 | 1. | – | – | 2.5 | – | – | – |
| 42C | – | Bal | 2. | 16. | .2 | – | – | – | – | – | – | – |
| 43C, 43CNS | – | – | 80. | 20. | – | – | – | – | – | – | – | – |
| 43F, 43F-NS | – | – | 80. | 20 | – | – | – | – | – | – | – | – |
| 54 | – | – | – | – | – | – | – | – | – | 99.0 | – | – |
| 55 | – | – | – | – | – | – | – | 99.0 | – | – | – | – |
| 56F-NS | – | – | 99.3 | – | – | – | – | – | – | – | – | – |
| 63 | – | – | – | – | – | – | – | – | 99.0 | – | – | – |
| 63-NS | – | – | – | – | – | – | – | – | 99.0 | – | – | – |
| 70C-NS | – | – | – | – | – | – | – | – | – | – | – | – |
| 71-NS | Bal | 1. | – | – | 4. | – | – | – | – | – | 12. | – |
| 80-NS | – | – | 12. | 3. | – | – | – | – | – | – | – | – |
| 81-NS | – | – | 20. | 5. | – | – | – | – | – | – | – | – |

**TYPE ANALYSIS OF METCO CERAMIC POWDERS**

| METCO Ceramic Powders | Aluminum Oxide | Zirconium Oxide | Titanium Dioxide | Calcium Oxide | Silicon Dioxide | Iron Oxide | Magnesium Oxide | Chromium Oxide | Others |
|---|---|---|---|---|---|---|---|---|---|
| 101-NS | Bal | – | 2.5 | – | – | – | – | – | – |
| 102 | – | – | 99.0* | – | – | – | – | – | – |
| 105-NS | 98.0* | – | – | – | – | – | – | – | – |
| 106-NS | – | – | – | – | – | – | – | 99.0 | – |
| 110 | 50. | – | 50. | – | – | – | – | – | – |
| 201, B-NS, NS | .5 | 93. | – | 5. | .4 | – | – | – | Bal |
| 210 | – | Bal | – | – | – | – | 24 | – | – |
| 211 | – | Bal | – | 31. | – | – | – | – | – |
| METCO Cermet Powders | | | | | | | | | |
| 410* | 70% METCO 101–30% METCO 404 | | | | | | | | |
| 411* | 30% METCO 101–70% METCO 404 | | | | | | | | |
| 412* | 65% METCO 101–35% METCO 404 | | | | | | | | |
| 413* | 35% METCO 201–65% METCO 404 | | | | | | | | |

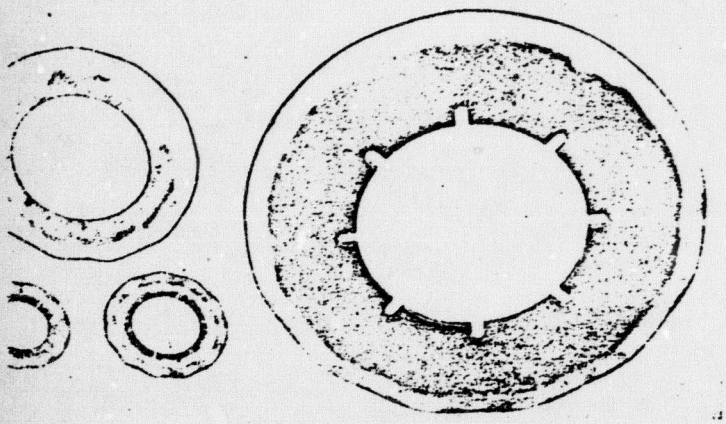
*Patent Pending
**Has Surpassed AMS 4775 in Many Applications
††16C Covered by U.S. Pat. No. 2,875,043

## typical applications

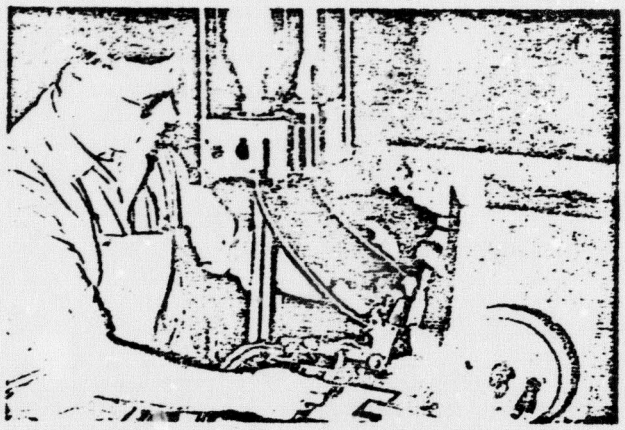
The ThermoSpray process has found wide application wherever service conditions are severe as, for example, in equipment for the petroleum and chemical industries. In the former it has been used with notable success on pump-plungers used in oil well pumping units and in pumps used in "water-flooding" which injects water into partially depleted oil pools to force out the remaining oil. Both the crude oil and salt water used in secondary recovery projects contain highly corrosive chemicals which would ruin ordinary metals in a short time. The ThermoSpray facings not only are highly corrosion resistant, but are also highly resistant to abrasion encountered in such service. The plungers range in size from 1 to 6 inches in diameter, 10 to 30 inches in length. Metco ThermoSpray Powder 15C or 16C is generally used on this type of part.

### Dry "Fluid" Drive

Dodge Manufacturing Company produces a unique dry "fluid" drive called Flexidyne. Instead of the usual fluid this device uses steel shot, which provides



Flexidyne rotors after being hard-surfaced by the ThermoSpray Process.



Two Metco ThermoSpray guns coating Flexidyne rotor blade. Though operator is shown with his hands on the gun they are actually mounted on a special fixture. Blade is rotated as coating is applied. Material is fed from hoppers suspended above.

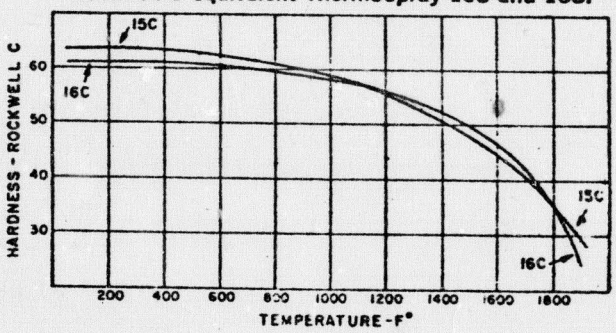
a number of advantages, one of which is 100% efficiency under full load speed. Heart of the drive is the rotorblade which must take a great deal of punishment from the steel shot. Before using ThermoSpray the company was paying $1.30 a pound for steel of sufficient hardness to resist the shot abrasion. This steel had to be worked hot and then annealed. With the ThermoSpray process and a self-fluxing alloy

**DENSITY OF CERAMIC COATINGS**

| ThermoSpray Coating Material | Coating Type | Density of Coating gm/cc | Weight of Coating | |
|---|---|---|---|---|
| | | | per sq. ft. Pounds | per .010" thick Grams |
| 101 | Soft | 2.61 | .136 | 61.5 |
| 101 | Hard | 3.06 | .159 | 72.2 |
| 105 | Soft | 2.62 | .136 | 61.7 |
| 105 | Hard | 2.9 | .151 | 68.4 |
| 201 | Soft | 3.64 | .189 | 85.8 |
| 201 | Hard | 4.29 | .223 | 101.3 |

**HOT HARDNESS**

**Rockwell C equivalent ThermoSpray 15C and 16C.**




**THERMAL PROPERTIES**

| ThermoSpray Coatings | Melting Point | Color | Thermal Shock Resistance | Thermal Conductivity | Total Emissivity | Thermal Expansion |
|---|---|---|---|---|---|---|
| 101 | 3700°F | Gray | Very Good | 19 BTU/hr/sq ft/in /°F at 1000-2000°F | .3-.4 | 4.1 x 10-6/°F 70°-2700°F |
| 105 | 3700°F | White | Very Good | 19 BTU/hr/sq ft/in /°F at 1000-2000°F | .3-.4 | 4.1 x 10-6/°F 70°-2700°F |
| 201 | 4650-4700°F | Tan-Yellow | Very Good | 8 BTU/hr/sq ft/in /°F at 1000-2000°F | .3-.4 | 5.4 x 10-6/°F 70°-2250°F |

containing a large percentage of tungsten carbide the company now deposits a "shot resistant" hard surface on a steel obtainable at only 7 cents a pound. which is softer and more shock-resistant.

## miscellaneous physical properties

### Fused coatings

Although these alloys melt at approximately 1900°F. they retain a relatively high degree of hardness at temperatures close to their melting points. This property combined with high resistance to oxidation and erosion make ThermoSpray 15C and 16C very useful on mechanical components subject to high operating temperatures.

### Tensile Strength

Tensile strength of ThermoSpray 12C, 15C, 16C, 17F are between 65,000 and 75,000 psi.

### Ceramic coatings—Hardness

The hardness of *grains* of alumina ($Al_2O_3$) and zirconia ($Zr\ O_2$) is well known. But the "hardness" of sprayed coatings of these materials varies a great deal with different spray techniques. For "soft" coatings, traverse speed, powder feed rate, and distance from nozzle to work are kept at a maximum and work temperature at a minimum. Opposite factors produce "hard" coatings. The former are more porous than the latter, therefore capable of more flexure and are much more resistant to failure by thermal shock.

The unique characteristics of high melting-point refractory materials such as zirconia and alumina has led to considerable interest in their use as thermal barriers, for wear-resistance, and corrosion-resistance to highly reactive materials. Such applications have included the protection of jet-starter housings during the high-temperature starting cycle and rocket engine mounting rings for protection during blast-off. Their excellent corrosion-resistance has led to many appplications on pump rods, plungers and seals, particularly in the chemical industry.

### Other recommended applications for Self-Fluxing ThermoSpray Powders

| Part: | Powder: |
|---|---|
| Pump Plungers | 12C, 15C, 16C |
| Crusher rolls | 16C, 32C |
| Pulverizing equipment parts | 15C, 16C, 32C |
| Glass molds | 12C, 15C |
| Heat treating rolls, etc. | 16C |
| Oil well pump plungers, polisher rods, etc. | 15C, 16C |
| Paper core winding mandrels | 15C, 16C |
| Pump sleeves | 15C, 16C |
| Conveyor screws, bearing housings | 15C, 16C |
| Steel mill roller guide boxes | 16C |
| Valve plugs | 12C, 15C, 16C |
| Plug gages | 15C |
| Farm implement parts | 15C, 16C |
| Mixing Machine blades | 15C, 16C |
| Textile spindles | 15C, 16C |
| Sizing mandrels | 15C, 16C |
| Abrasive wheel molds, plungers | 15C, 16C |
| Diesel engine valves, stems | 15C, 16C |
| Valve stems | 15C |
| Extrusion rams | 16C |
| Wire drawing capstans | 16C |
| Concrete block cores | 16C |

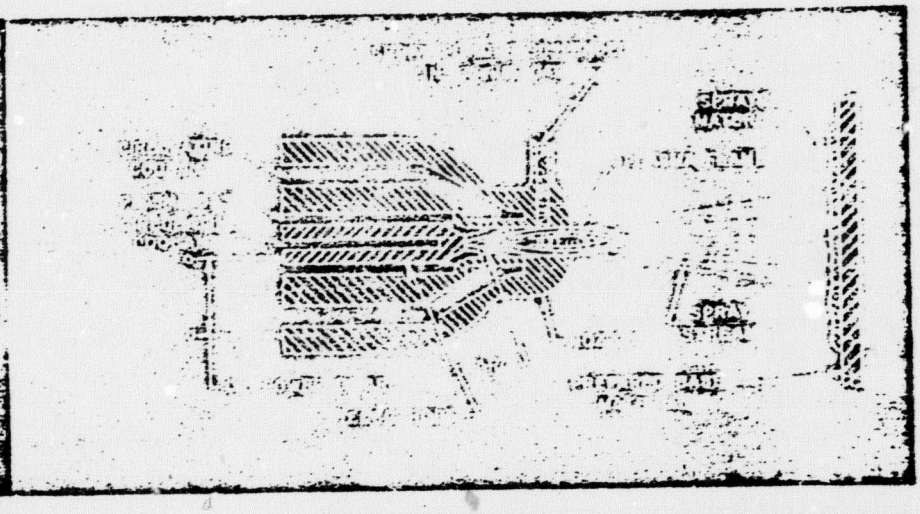
### Other recommended applications for Tungsten Carbide ThermoSpray Powders

| Part: | Powder: |
|---|---|
| Pulverizing equipment parts | 31C, 32C |
| Conveyor screws, housings | 31C, 32C |
| Buffing fixtures | 31C, 32C |
| Sizing punches, pilots | 32C |
| Plug gages | 32C |
| Farm implement parts | 31C, 32C |
| Mixer blades | 31C, 32C |
| Extrusion mandrels | 32C |
| Abrasive wheel molds, plungers | 32C |
| Wire drawing capstans | 31C, 32C |

### Other recommended applications for Ceramic ThermoSpray Powders

| Part: | Powder: |
|---|---|
| Nose Cones | 101, 201 |
| Pump plungers | 101 |
| Dental crucibles | 101 |
| Graphite crucibles | 101, 105, 201 |
| Heat treating equipment | 101 |
| Casting inserts | 101 |
| Jet engine starters | 101 |
| Missile parts | 101, 201 |
| Pump sleeves, face plates | 101 |
| Metal pouring troughs, spouts | 101 |
| Annealing rolls | 101 |
| Seal rings | 101 |
| Trays, boats, etc., for sintering | 101 |
| Cutting lances, flame brooms, nozzles | 105 |
| Lapping plates | 101 |
| Permanent mold facings | 101 |
| Galvanizing kettles | 101 |
| Plug gages | 101 |
| Jet engine parts | 101, 105, 201 |
| Rocket nozzles | 101, 201 |
| Gas turbine parts | 101, 105, 201 |

# THE METCO PLASMA FLAME SPRAY PROCESS— HOW IT WORKS



The Metco Plasma Flame Spray Gun utilizes an electric arc contained within a water-cooled jacket. An inert gas, passed through the arc, is "excited" to temperatures up to 30,000° F. The plasma of ionized gas issuing from the torch resembles an open oxy-acetylene flame in shape and appearance.

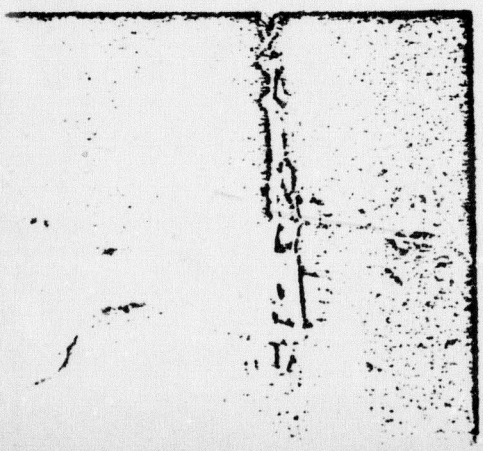
The Metco Plasma Flame Gun operates on inexpensive polyatomic gases, with high electrical power conversion efficiency and long component life. Continuous hot gas streams can be generated for costs of 1/3 to 1/2 that of an oxygen-fuel gas flame of equivalent heat output. In some applications closed-circuit systems can be arranged permitting recovery and re-use of high percentages of the gases sprayed. Attendant advantages are long component life, push-button operation and elimination of flashback and explosion problems.

## materials that can be sprayed

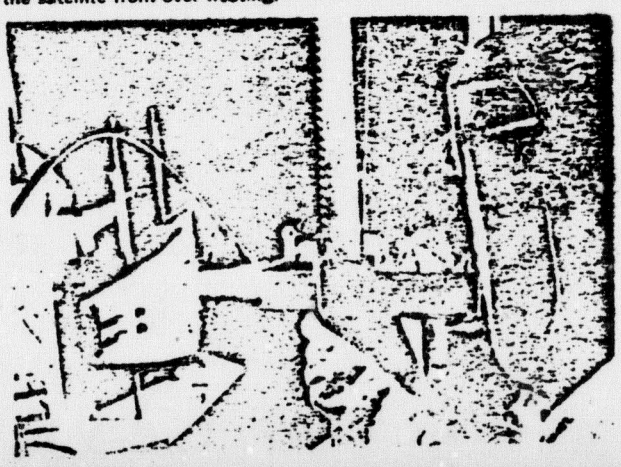
The materials listed on the right and mixtures thereof have been sprayed with the standard apparatus on various shaped objects and mandrels:

| Materials | Melting Point F° |
|---|---|
| Aluminum Oxide | 3772 |
| Calcium Zirconate | 4253 |
| Chromium | 3430 |
| Chromium Carbide | 3434 |
| Ceric Oxide | (approx.) 4712 |
| Cobalt | 2723 |
| Cobalt-Zirconia (blend) | 2723-4892 |
| Magnesium Zirconate | (approx.) 3848 |
| Molybdenum | 4748 |
| Nickel | 2650 |
| Nickel-Alumina (blend) | 2650-3772 |
| Titania-Alumina (blend) | 3344-3772 |
| Titanium Oxide | 3344 |
| Tungsten | 6170 |
| Tungsten Carbides | 5198 |
| Zirconium Oxide (stabilized) | 4712 |
| Zirconium Silicate | (approx.) 4388 |

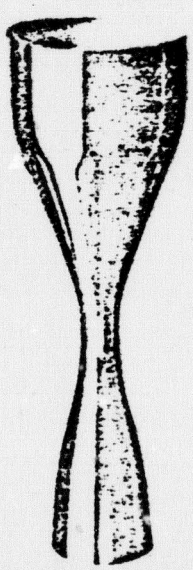
Graphite missile thrust insert is plasma coated with tungsten, .125 inch thick.



Telstar shell is plasma flame coated with aluminum oxide. Coating will reflect or reradiate energy better than most materials, thus help keep the satellite from over heating.



Venturi throat for rocket engine. Aluminum mandrel, machined to exact contour of throat was flame sprayed with zirconia. Outer layer of nickel applied and mandrel leached out with acid.



Physical and metallurgical properties of the coatings are generally superior to conventional flame-sprayed coatings. These include reduced porosity, improved bond and tensile strengths, less oxide content in the case of metal and high density. Coating densities run up to 98% of theoretical and are easily controlled. These factors will undoubtedly widen the industrial use of plasma sprayed coatings. For example, pure tungsten and tungsten carbide can be applied to almost any base material with densities of about 95%.

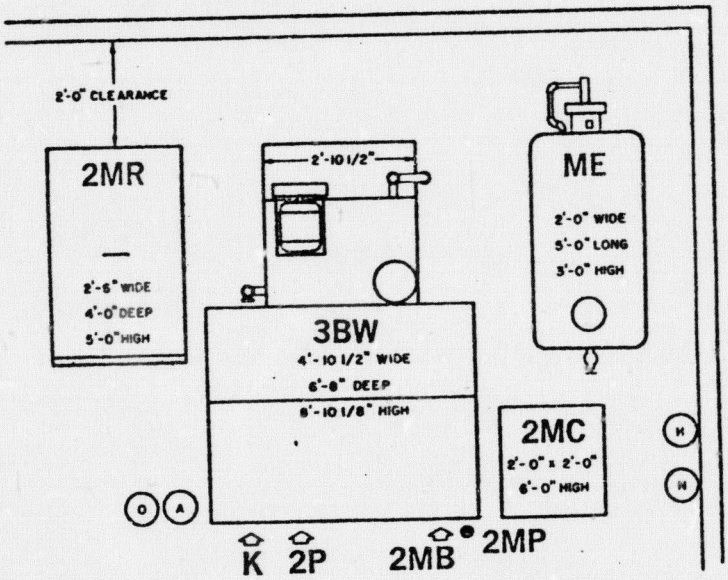
## other materials that can be sprayed

In general most inorganic materials that can be melted without decomposition can be applied. Others now in development work include—tantalum, palladium, platinum, zirconium diboride; columbium, hafnium, vanadium carbides and refractory borides of hafnium and cerium.

## applications

The very great range of operating temperatures that can be produced, the variety of materials that can be sprayed, and the basic economies of the process would seem to limit possible applications only to the imagination of the designer and research development engineer. Applications already used include the spraying of rocket nozzles and nose cones and other parts subject to extreme thermal conditions, such as crucibles for high melting point materials. In the electronic industry parts for large vacuum tubes have been successfully coated and in the chemical field considerable work has been done with catalytic as well as protective coatings.

## floor space requirements for typical research department installation




| | | | |
|---|---|---|---|
| K | Wire Metallizing Machine | 2MR | Plasma Flame Power Supply Unit |
| 2P | ThermoSpray Powder Gun | MS | Safety Unit (gloves etc. not shown) |
| 2MB | Plasma Flame Spray Gun | 3BW | Water Wash Spray Booth |
| 2MC | Plasma Flame Control Unit | A | Acetylene |
| ME | Plasma Flame Heat Exchange Unit | H | Hydrogen Supply |
| 2MP | Powder Feed Unit | N | Nitrogen Supply |
| | | O | Oxygen |

## handbook

A comprehensive 320-page volume covers all phases of modern metal and ceramic spraying (except the new Plasma Flame Process.) Over 150 diagrams, photos, charts and tables. An invaluable reference for anyone concerned with metalworking.
Price $5.00 clothbound.

## further information

The following specialized bulletins are available and will be made on request.

Bulletin 51—General round-up of more than two dozen typical maintenance applications with photographs and complete data. 8 pp.

Bulletin 93—On sprayed metal/paint systems for long term (10-50 years) corrosion protection.

Bulletin 142—Describes the Type MB Plasma Flame Spray Gun and how it works. Also lists applications and materials that can be sprayed. 8 pp.

Reprint 142—Metallizing and its Application in Aircraft Gas-Turbine Components—a most informative article by Donald E. Hacker of The General Electric Co. It is a reprint from the WELDING JOURNAL.

Reprint 144—An article reprinted from INDUSTRIAL LABORATORIES "Coatings Boost Metals Into High Heat Ranges" by John Long, Director of Research, Solar Aircraft Co. 6 pp.

Bulletin 153—The ThermoSpray powder process for applying nickel-chromium hardfacing alloys, tungsten carbides, METCO 404 nickel aluminide, alumina, zirconia, stainless steel and other materials. 8 pp.

## engineering assistance

In general it is recommended that due to the special circumstances surrounding any particular application, experimental work be carried out since both design data and actual production line operations will vary considerably. Metco and its engineering staff stand ready to assist you in such work as well as provide any available additional information that is desired.

You are cordially invited to visit Metco's modern plant at Westbury, Long Island, to discuss your problems with our personnel, or if you prefer, we will arrange to have a field engineer address group meetings of interested individuals at your own plant

PLAINTIFF'S EXHIBIT
HAW 91 Evd
7-26-02

E1062

Maximum Oxygen

19 Dec 73

$Al_2O_3$

| | $Al_2$ | $O_3$ |
|---|---|---|
| Mol Wt | 27 | 16 |
| | ×2 | ×3 |
| | 44 | 48 |

Assume equal wts. (in grams)

Require 5 grams $O_2$ to react with 5 grams Al.

Argon with 5 ppm oxygen

10 cubic ft/hr flow × 6 min (0.1 hr)

= 1 cu ft

× $(2.5 \times 12)^3$ cc/cu ft (≈30,000

= 30,000 cc

÷ $\frac{5}{1,000,000}$ = $\frac{150,000}{1,000,000}$ = 0.15 cc ($O_2$)

× 1 (= 0.001 gm/cc)

= 0.00015 grams $O_2$ $\frac{.00015}{5} = .003\%$

Treated aluminum powder:

Surface area of 1 gram ≈ 4.0 sq m/gm (or ≈ 8 sq m/gm Tabs

$\frac{100 \text{ sq m/gm}}{8 \text{ sq m/gm}}$ = 25 cc; half in air, i.e. 12 cc (net

20% $O_2$; 10 cc × 20% = 2 cc ($O_2$)

2 cc × 0.001 gm/cc = 0.002 grams $O_2$ $\frac{.002}{5} = .0$

Monthly indoor, flow is 0.5 gm/gm powder

From $CO_2$, (+ wt C = 12, O in it, each gram

) gram = 2× wt of O oxygen Basis could be 1.0

E1063

PART NO. 990092

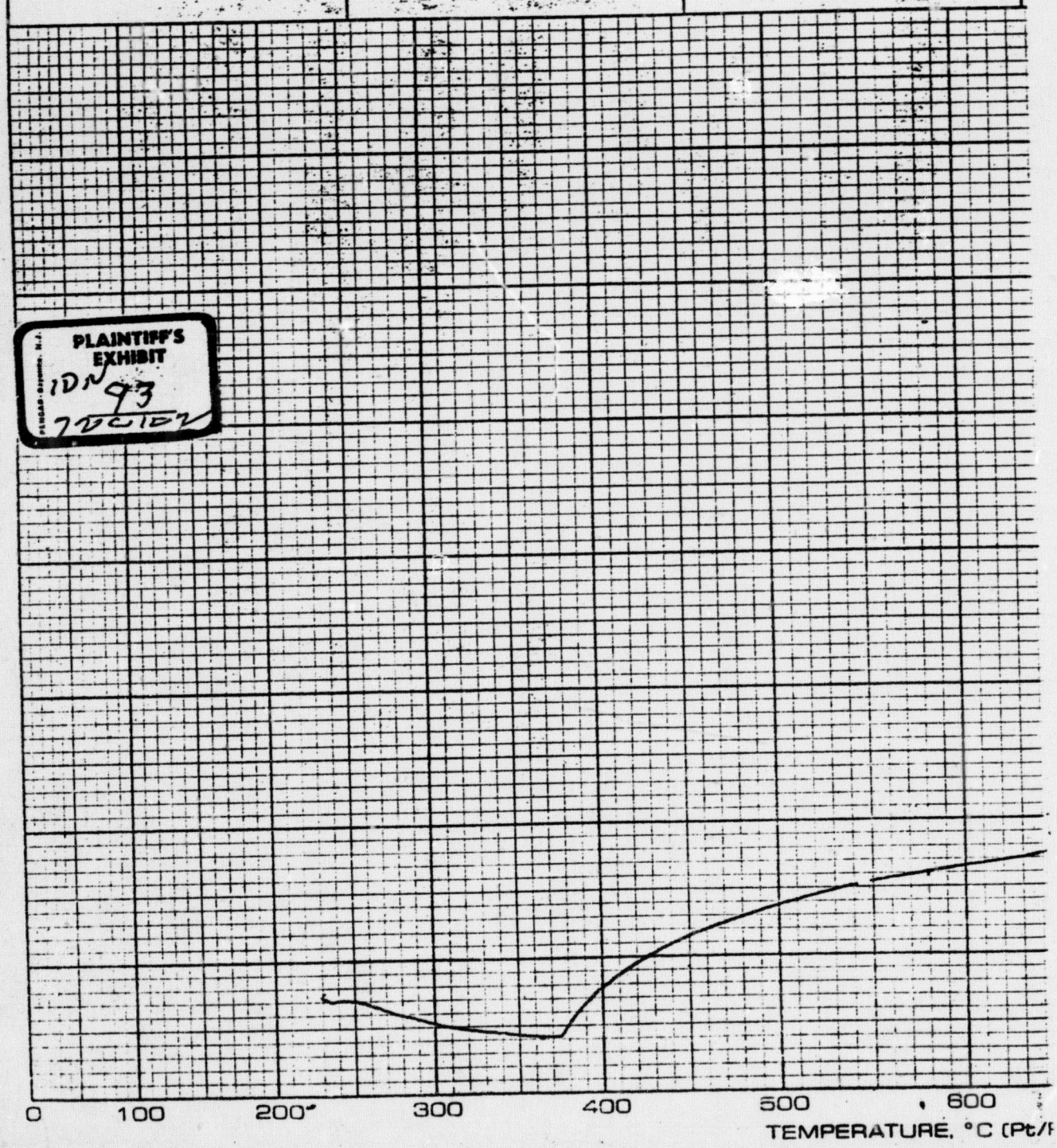
E1064

| | | |
|---|---|---|
| RUN NO 1 DATE 11/30/73 | T-AXIS | DTA-DSC Sample |
| OPERATOR S.J. BERMAN | SCALE, mv/in 0.8 | SCALE, °C/in |
| SAMPLE: 80/20 - N./AL | PROG. RATE, °C/min 50 | (mcal/sec)/in |
| ATM AIR @ 1ATM | HEAT X COOL ISO | WEIGHT, mg 82.5 mg |
| FLOW RATE STILL | SHIFT, in 0 | REFERENCE Al2O3 |

DUPONT Instruments

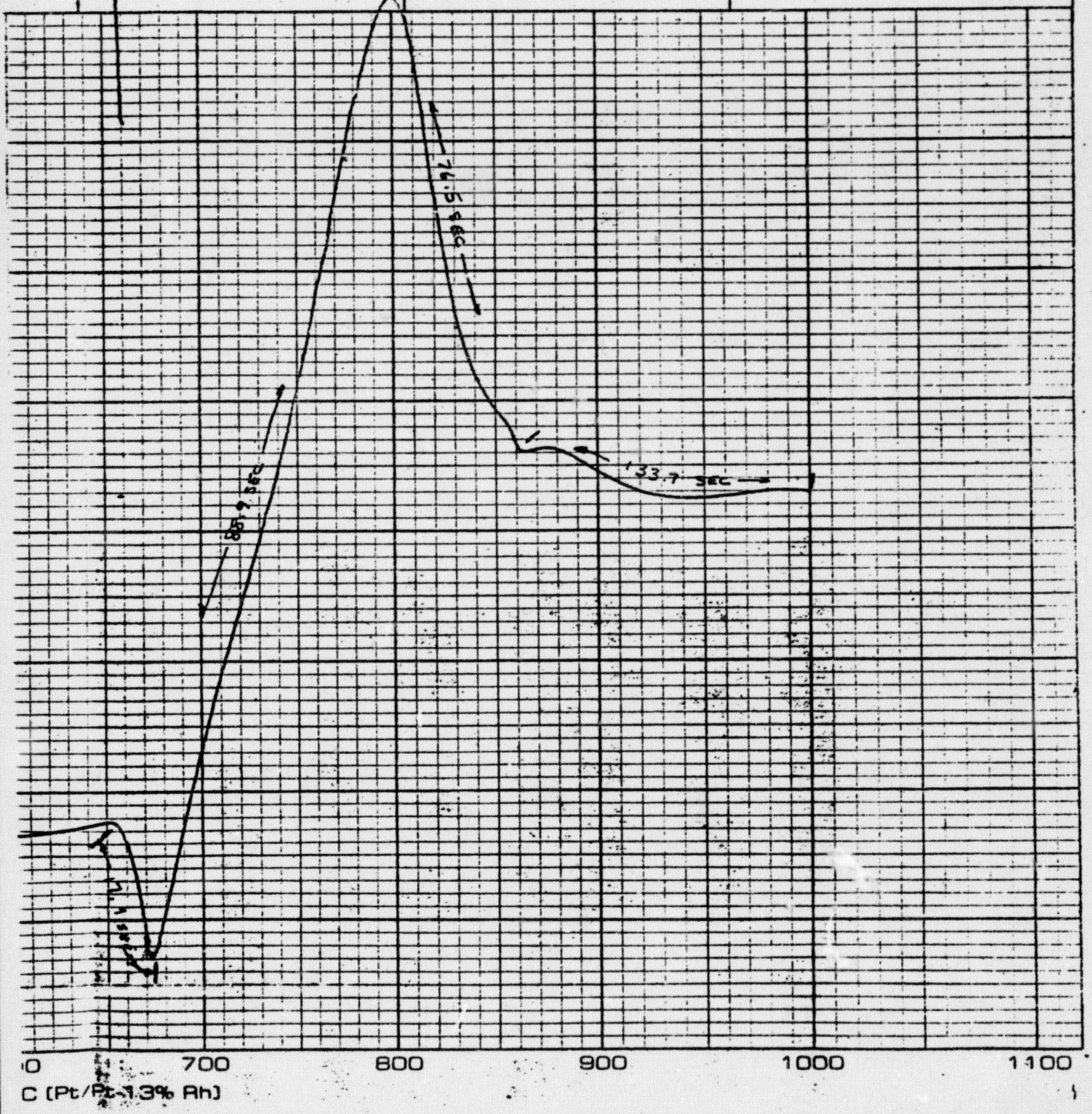
MEASURED VARIABLE

PLAINTIFF'S EXHIBIT
IDN 93
7DC10



E1065

| | TGA | TMA | |
|---|---|---|---|
| | SCALE, mg/in | SCALE, mils/in | Heating 90 |
| | SUPPRESSION, mg | MODE | Cooling 0.2 |
| g | WEIGHT, mg | SAMPLE SIZE | |
| | TIME CONST., sec | LOAD, g | |
| | dY (mg/min)/in | dY (10X), (mils/min)/in | |



PART NO. 990092

E1066

RUN NO 3 DATE 11/30/73

OPERATOR S.J. BERMAN

SAMPLE: EUTECTIC 95/5 #29904

ATM AIR @ 1 ATM

FLOW RATE STILL

T-AXIS

SCALE, mv/in 0.8

PROG. RATE, °C/min 50

HEAT X COOL ISO

SHIFT, in 0

DTA-DSC

SCALE, °C/in 5 green

(mcal/sec)/in

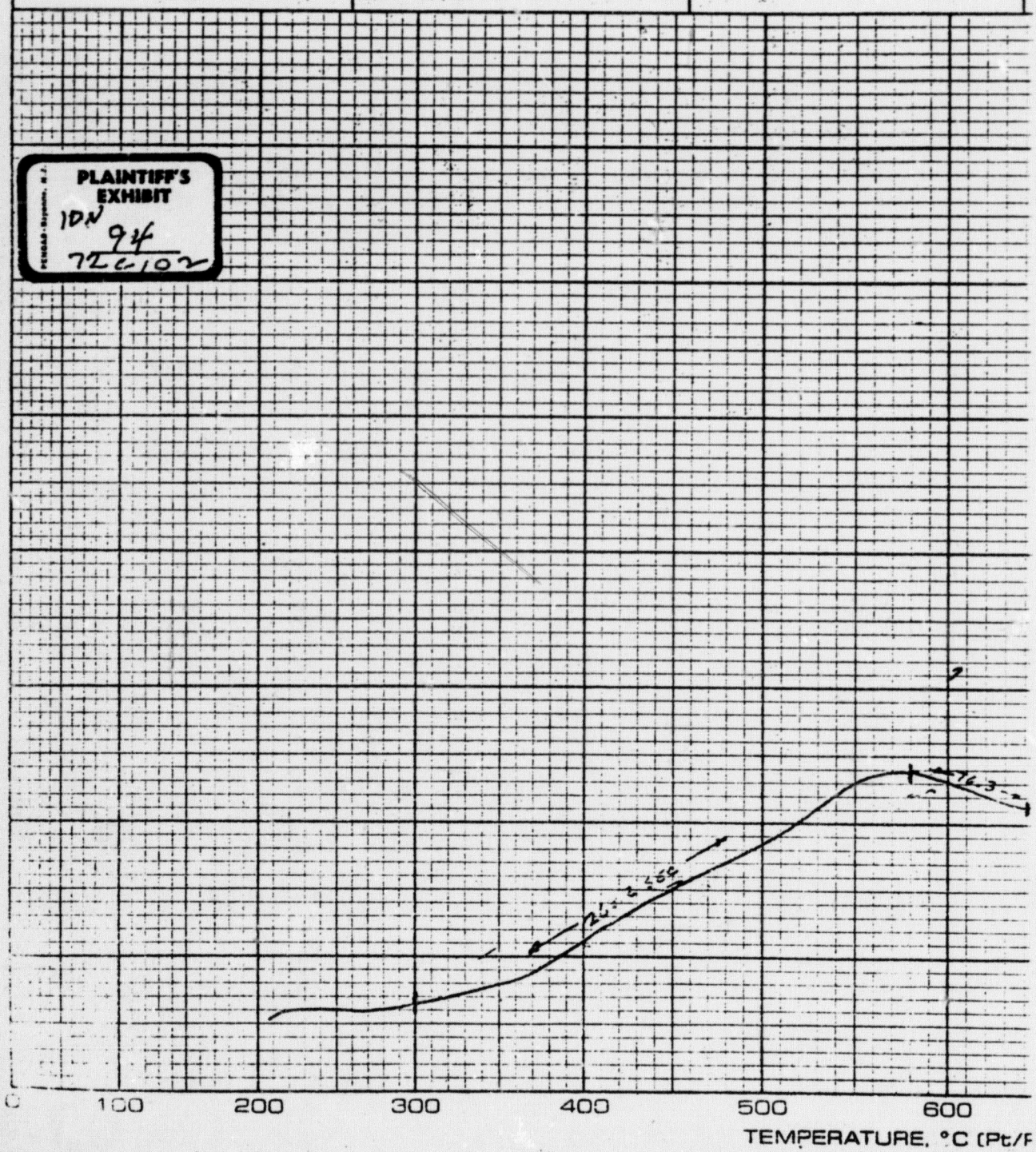
WEIGHT, mg 92.5 mg

REFERENCE $Al_2O_3$

PLAINTIFF'S EXHIBIT
IDN 94
72-C-102

DU PONT Instruments

MEASURED VARIABLE



E1067

| | TGA | TMA | Heating 1° /min = .6°/sec<br>Cooling about 0.1 – 2.5°/sec |
|---|---|---|---|
| | SCALE, mg/in | SCALE, mils/in | |
| | SUPPRESSION, mg | MODE | |
| 15 | WEIGHT, mg | SAMPLE SIZE | |
| | TIME CONST., sec | LOAD, g | |
| | dY, [mg/min] /in | dY, [10X], [mils/min]/in | |

PX-95

E1068

E 1069

# EXCERPTS FROM DEPOSITION OF FERDINAND J. DITTRICH

Dietrich

* * *

in the art at the time.

Q Let us do that. Was nickel as a flame spraying powder known in the world in the period 1951 through 1961?

A It may have been known. To my knowledge, it wasn't generally used.

Q Was it a commercially available flame spraying powder in that period of time?

A No.

MR. SPRUNG: You are talking about pure nickel now?

MR. CALIMAFDE: Nickel.

A No, to my knowledge it wasn't.

Q Was aluminum available as a flame spraying powder in that period of time?

A There were a number of aluminum powders available and some of them could be sprayed with some of the equipment that was available at the time, the Schori equipment for instance, that was capable of spraying an aluminum powder but not very effectively.

Q Do you know whether nickel wire was used in the flame spraying process during that same period of time 1951 through 1961?

A Nickel wire was available as a flame spray wire,

* * *

E 1071

# EXCERPTS FROM DEPOSITION OF JOSEPH F. QUAAS

you could provide me with the most information as to the composition and form of these materials and their development, is that correct?

A To some extent, perhaps.

Q Do you know of anyone who might have more information concerning these materials or their development?

A Probably not.

Q Let's get to the material called ExoTex. That has a number designation also.

A 29904.

Q When did Eutectic first start marketing this material, to your knowledge?

A I don't remember.

Q I hand you a copy of Defendant's Exhibit 3 and I will ask you if this might refresh your recollection as to the period of time that Eutectic might have first started manufacturing and selling the ExoTex type material.

A Yes.

Q What is the date of that letter?

A July 10, 1970.

Q From that letter, can you give me a date how much earlier they might have first sold it?

A No, I can't.

Q You have no recollection?

A No.

Q Was it roughly within this time period?

A I would say so.

Q Within a month or so?

A Well, I am not sure of that.

Q Two months?

A I don't know.

Q What isyour best estimate of the earliest period prior to this letter?

A At the most, I would say six months.

Q The ExoTex 29904, as sold by Eutectic, was that material sold prior to the commencement of this lawsuit?

A Yes.

Q And it is being presently sold?

A Yes.

Q Is this material a thermo spray or flame spray powder?

A It's a flame spray consumable.

Q Powder?

A Yes.

Q Do the particles have a nickel core?

A Yes, sir.

Q Do they have a coating on that core?

A Yes, sir.

Q Is that coating a coating of aluminum?

A Yes, sir.

Q Bound to the core by a binder?

A Yes, sir.

Q Can you tell me approximately what the particle size or particle size range of the nickel core is?

A I would say minus 140 mesh, ~~minus~~ plus 325.

Q In other words, it will all pass through a 140 mesh screen?

A No, I didn't say that. There can be a few percent above the 140 screen.

Q And that's substantially pure nickel, the core?

A Yes.

Q What type of coating material, pure aluminum?

A Yes, sir.

Q Aluminum powder?

A Yes, sir.

Q What is the particle size of the aluminum?

A I don't know.

Q Very fine?

A Very fine.

Q Minus 325 mesh?

A I wouldn't guess.

Q Could you determine that for me and let me know?

A Sure.

Q Would you supply that?

A Yes.

Q What type of binder do you use?

A As I recall, it's a resin.

Q Do you have any idea of the composition ?

A No.

MR. KAPRALOS: This is under the protective order.

MR. CALIMAFDE: Is this confidential?

MR. KAPRALOS: The binder is definitely.

MR. SPRUNG: When you get the deposition, you can mark that.

MR. CALIMAFDE: Are you about to disclose the nature of the binder?

THE WITNESS: No.

Q Would you find out what the nature of the

binder was?

A How do you want this description?

Q If you would say something like phenolic varnish, that would be--

A All right.

Q IS it a phenolic varnish?

A I am not sure.

Q In making this powder do they disperse the fine aluminum in the binder and then coat the nickel particles?

MR. CALIMAFDE: Is that confidential information?

THE WITNESS: I think so.

MR. SPRUNG: Off the record.

(Discussion off the record.)

MR. CALIMAFDE: The record should be clear the information you are about to obtain from the witness is considered confidential and comes within the scope of the protective order.

A The product can be manufactured two ways.

Q I am asking you how it is manufactured presently.

A I said two ways.

Q It is presently manufactured two ways?

A One, yes, the aluminum powder is introduced into a slurry. The second way is to introduce the aluminum powder separately from the slurry.

Q Let's go to the first. You sort of make an aluminum paint so to speak of the varnish?

A Yes.

Q And then coat the nickel particles with it?

A Yes.

Q And the second way?

A The second way, the mixing operation is performed in a different type of mixer, and under those circumstances, the aluminum is added to a nickel powder slurry, pre-mix.

Q In other words, in the first method you put the aluminum in a binder, make sort of a paint and put it on the nickel?

A Yes.

Q In the second, you mix the nickel powder with the binder and add--

A Correct.

Q But in each case you wind up with a nickel core coated with an aluminum--

A Bound aluminum powder.

Q About how much percent aluminum as compared to the nickel?

A Five, by weight.

Q About five percent by weight. Could you briefly tell us historically how Eutectic came about to develop this powder, 29904 powder?

A We have in our presence an individual who took it upon himself, while, I believe I was in Europe, or some other God-forsaken place, to assign this to an employee of our company, from a project point of view.

Q Would you further explain who the individual was?

A Mr. Kapralos.

Q About what time did he do this?

A I can't remember the exact date.

Q Did he discuss it with you ahead of time?

A No.

Q This ExoTex 29904, is intended for use in a flame spray process, is that correct?

A Correct.

Q In that process you feed the powder into a flame spray gun?

A True.

Q And you heat it up, it is melted or heat softened and you project it on a surface to be coated?

A Correct.

Q Say it's a metal surface, steel surface or a shaft, do the individual particles bond tothe surface?

A Yes, sir.

Q What kind of bond do they form, a metallurgical bond, like a little spot weld?

A There is a micro bond phenomenon that occurs, ~~articled at the substrate~~.

Q Is there an exothermic reaction when this material is sprayed?

A Yes.

Q And that developes heat, aiding in the bonding?

A Yes, sir, similar to a thermit reaction.

Q Exothermic reaction?

MR. CALINAFDE: I think the witness said similar to a thermit reaction and you said exothermic reaction.

Q Is it an exothermic reaction?

A Yes.

MR. KAPRALOS: He said --

MR. SPRUNG: Would you let one attorney please conduct this examination. You had your opportunity, Mr. Kapralos.

Q So there is an exothermic reaction as the material is sprayed, generates heat and it aids in bonding to the surface?

A Yes.

Q Are you familiar with the flame spray process, in general, what is known as flame spraying?

MR. CALIMAFDE: As of now?

MR. SPRUNG: Yes.

A Generally.

Q Would you describe what that process is?

A It is the application of, usually, powder type consumables that are fed, ultimately, into a flame which naturally imparts heat to the powder consumable during it's transfer from the, let's say, ~~type~~ tip of torch to a given substrate.

Q Are you familiar with flame spraying of wires or rods?

A To some extent, yes.

Q And in each of those processes, the

A Correct.

Q And that was back in '53?

A In '52 or '53.

Q From that time on, up until the time you started working on the predecessor of the ExoTex 29904, you did no further work in this field?

A That's correct.

Q Do you know what instigated you to start work on the Exothermic materials?

A Mr. Kapralos.

Q Did he ever discuss the project with you or why he wanted to do this?

A No, sir.

Q Were you familiar with any commercial products sold by anyone at the time this product--

A Not at that time, no.

Q You had no knowledge?

A No.

Q Other than the nickel aluminum composites, exothermic composites, do you know of any other self-bonding material in the flame spray field?

A Moly.

Q When you say moly, do you mean molybdenum?

* **

MR. SPRUNG: We are going to reach a point where I am going to have to move for answers.

Q Have you been apprised of what has been going on in this suit?

A Superficially.

Q Just superficially?

A Yes.

Q Have you been sent regular reports?

A No.

Q Has your advice been asked on any of the technical matters?

A No.

Q Would you briefly tell me the present organizational set-up at Eutectic, who is in charge, the various departments and who reports to who?

A We have three divisions within a world-wide organization. One is international, one is European--

Q Would you give us the names?

A One is North America, which includes the U.S.A. and Canada. At the head of the North America Division is Mr. Paul Albright, who is president, and he has reporting to him a marketing head, technical head and control area head, fiscal. Fiscal is Mr. Starkowsky, technical is myself and marketing is

Mr. Hammond.

Q This is the U.S. Eutectic Corporation?

A This is North America Division.

Q Of Eutectic who are the plaintiff in this action?

A I believe so.

Q Who is head of the whole organization?

A We have a chairman, Mr. Frank Glaser.

Q Where is he located?

A He is located in Flushing.

Q Do you have any other divisions besides the North American Division?

A The European Division who is headed by their president, Mr. Gigon.

Q Is that under the name Eutectic also?

A No, I believe it's under the name Castolin.

Q What is the relationship between Castolin and Eutectic?

A Castolin is the name, the tradename that has been applicable for the European Division for God knows how many years, ever since I have been with the company.

Q But it is all one company?

A The ownership and details such as that, am not aware of.

I

* * *

the test sample.

Q Were any detrimental effects noted by adding the lead chromate to the powder?

A Not to my knowledge.

Q Any increasing fuming?

A I can't answer that question.

Q How did the DuroTec development come about?

A What the hell is DuroTec?

MR. SPRUNG: Off the record.

(Discussion off the record.)

Q Can you tell us what DuroTec 19910 is?

A I believe it's a nickel base alloy, containing nickel base, silicon, boron, self fluxing, naturally, and I think the product consists of 90 percent of that item and ten percent of the 29904 material.

Q In other words, it is a physical mixture of conventional type self fluxing nickel base alloy powder with the ExoTec 929904 powder?

A Right.

Q What is the purpose of adding the ExoTec 29904 to the self fluxing nickel base alloy powder?

A To afford better bond, as sprayed.

* * *

exothermic bonding materials about 75 percent that you sell are Xuper Bond and 25 percent the ExoTex?

A Yes, 29904.

Q Are there any applications on which you can't use the Xuper Bond but can use the ExoTex?

A Not to my knowledge.

Q In other words, they are equivalent?

A Yes, except that the Xuper Bond provides the higher strength levels.

Q And is yellow?

A By the time you get through with it it is yellowish green.

Q I am handing you a document which previously has been marked as Defendant's Exhibit 14 and it is dated 1-8-70. Can you identify that document or tell us anything concerning its subject matter?

A I can't. These identifications do not at all ring a bell.

Q You have no idea what Spram Pre Bond powders are?

A No.

Q I hand you Defendant's Exhibit 16 for identification. Does that refresh your recollection

concerning Spram Pre Bond powders?

A I am sorry, but these are not identified and I don't remember that they were, and particularly in view of the fact I see they were compared to 404.

Q It does indicate that the tests were requested byyou, however?

A Sure.

Q I hand you Defendant's Exhibit 15 for identification.

A Now they are identified, nickel oxide aluminum combinations.

Q Does that refresh your recollection as to any work done on nickel oxide aluminum powder samples?

A Apparently yes. I have to say yes.

Q Will you tell us what you recall about this type of powder?

A I do[illegible]remember how it was made up but apparently the two raw materials involved are nickel oxide, which I assume is in powder form and, naturally, the aluminum powder.

Q Do you recall the purpose of making these powders and testing them?

A I would imagine, to evaluate whether these

types of materials would be satisfactory for spray purposes.

Q Why were you comparing it with Metco 404?

A I can't say.

Q You have no recollection of any attempt to find something that was equivalent to Metco 404?

MR. CALIMAFDE: I don't like your use of the word equivalent. That has a legal connotation as well as a lay connotation.

Q To be competitive with Metco 404.

A I would say it would be maybe in the ball park. When you say competative, I am thinking of pricing as well.

Q What is the purpose,to find a bonding powder which Eutectic could market for bonding purposes, the same way that Metco marketed the 404 powder?

A Yes.

Q And do you know why nickel oxides were used?

A The only thing I can recall on the nickel oxide, I believe that it was explored as a source of nickel in melting, and atomizing nickel base alloy powders.

Q Do you have any recollection of work done at Eutectic in an attempt to use an oxide for the reaction with the aluminum, rather than nickel, in order to avoid Metoo's patents?

A Nickel titanium.

Q What was that?

A I remember a powder of that type being test evaluated.

Q In order to avoid the nickel aluminum of Metco's--

A I wouldn't say that. I think it was out of sheer curiosity, technical curiosity.

Q Do you recall any work being done at Metco in an attempt to find the material which would avoid Metco's--

A Being done at Metco?

Q Being done at Eutectic to find the material in order to avoid Metco's patent on an aluminum intermetallic reaction?

A No.

Q I refer to Defendant's Exhibit 20 for identification, and I will ask you to read that document to see if it refreshes your recollection.

MR. CALIMAFDE: I don't see any reference

in that to the patent at all.

MR. SPRUNG: I am asking him if it refreshes his recollection.

Q I would refer to the--

A This is in line with a project Kapralos lodged with Yurasko.

Q I refer you to the last paragraph.

MR. CALIMAFDE: The word, "Claimed."

MR. SPRUNG: Yes.

Q I am asking you if that refreshes your recollection as to any work was done at Eutectic in an attempt to avoid Metco's patents.

A It indicates that.

Q Would you refer to the next page also.

MR. CALIMAFDE: You say this document indicates that?

THE WITNESS: Yes. He says, "In place of."

MR. CALIMAFDE: He is talking about patent.

MR. SPRUNG: Will you not coach the witness.

MR. CALIMAFDE: There is no point in confusing the record.

MR. SPRUNG: We are not confusing the record.

Q You received a copy of Defendant's Exhibit 20?

A Yes.

Q It is your understanding that the final objective as set forth in the last sentence on page one stating, "Final objective, indicative of samples 160-3 and 160-10 is to coat the nickel base powder, regardless of chemistry, with an oxide layer to react with aluminum via the aluminothermic reaction, as indicated below, in place of the pure intermetallic reaction as claimed by Metco."

Was it your understanding they were trying to develop a powder that would have the oxide aluminum in place of the intermetallic reaction claimed by Metco in its patents?

A I can't answer that. I don't know what 160-3 was.

Q I hand you Defendant's Exhibit 2 and refer to the sample number whih indicates a nickel oxide core material.

A I would say yes.

Q Do youknow if you were successful at Eutectic in developing a powder in which a nickel oxide core or a nickel core with an oxide layer was coated with an aluminum to provide an aluminum nickel oxide reaction?

A I do not.

Q Does Eutectic make any such powder?

A No.

MR. SPRUNG: I refer to document 50154, which I will ask the reporter to mark.

(Defendant's Exhibit 56 marked for identification, a document 50154.)

Q Was that written by you, Mr. Quass, on April 16, 1970?

A That's correct.

Q And did it refer to the previous exhibit, which we have just discussed, Exhibit 20?

A Apparently this letter is based on that report.

Q When you say, "I make reference to your letter of April 15th," were you referring to the report of April 15th, Defendant's Exhibit 20?

A I can't answer that. Usually I will make reference to a test evaluation report and not a letter.

Q Would you check your files and see if there is any letter?

A Yes.

Q Was the subject matter you were referring

* * *

successful in this lawsuit, then we will tell you.

Q Who is Dr. C. Vuilleumier?

A I would call him technical director of Castolin, located in Lausanne, Switzerland.

Q Did you at Eutectic demonstrate your RotoTec process?

A In the field you mean?

Q Yes.

A Yes.

Q When you demonstrate it, you take the ExoTex 29904 or the Xuper Bond, and flame spray it?

A Yes.

Q On a surface?

A Yes, sir.

Q And that involves passing the powder through a heating zone of a flame spray gun until it is melted or heat softened?

A Heat zone outside the spray gun.

Q The flame of the gun?

A Yes. You said through the gun. No, we don't do that. It doesn't pass through the gun.

Q I understand what you are saying. I apologize. The powder is sprayed, using the gun, and it is passed through the heating zone formed by

the gun, it is heated up, an exothermic reaction occurs and it is projected on a substrate where it forms a bond coat, is that correct?

A Yes, sir.

Q And Eutectic practices that process and demonstrates it?

A Yes, sir.

Q What is the R.W. Torch?

A The European version of the RotoTec Torch.

Q What is M.T.U. in Munich?

A I have no idea.

Q I hand you Defendant's Exhibit 48 and ask you if that gives you any indication?

A It says here it's a licensee of G.E., but I don't know the organization at all.

Q Who is Professor Doctor Knotek and Mr. H. Steine?

A Steine is a metallurgist at Castolin, and Doctor Professor Otto Knotek is now a top professor at the Aachen University in Germany.

Q At the Technical University in Aachen?

A Yes.

Q Has he any connection with Castolin?

A At the present time, I am not positive.

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Q Have you?

A On ExoTex?

Q Yes. I assume Xuper Bond is an ExoTex with an additive.

A Right.

Q Other than the coloring materials, did you try any other additives with ExoTex type materials?

A I believe so, but I can't recall what they were. I can state that they presumably would be in the same category as the lead chromate. In other words, they would be oxides that were conducive to providing additional oxygen in the flame. I think moly oxide was tried. I don't remember anything more than that.

Q Are there any experimental reports which indicate tests made with ExoTex materials containing any additives other than the dyes which you have produced?

A I think not. That was a formal project and under the circumstances there would be a report issued, a test report, which was the case.

Q So you have no other tests or reports in which ExoTex type material was sprayed and any in which additives were--

* * *

which I will ask the reporter to mark.

(Document 50049 marked Defendant's Exhibit 72 for identification.)

Q Can you identify that document?

A Yes, sir.

Q What is it?

A This is a communication from Mr. Patel to myself indicating that the Xuper Bond product does not comply with the 1337 spec.

Q That's the Pratt & Whitney spec?

A Yes.

Q Does that refresh your recollection whether the ExoTex does meet the Pratt & Whitney spec?

A I think it might, chemistry-wise, yes.

MR. CALIMAFDE: That question was not asked.

A (Continuing) From our point of view, I would say yes.

Q But the Xuper Bond does not because the spec has a certain limit on impurity content.

A Correct.

Q You testified, I believe, that Xuper Bond contained approximately one and a half percent of the lead chromate.

* * *

E 1096

# EXCERPTS FROM DEPOSITION OF JOHN PAUL BRODERICK

point?

A They became more involved when Mr. Marden relocated to California. I was put in charge of the laboratory.

Q About what time was that?

A Probably in '51.

Q From '49 through '51 your duties were pretty much the same?

A They became more involved on a daily basis essentially because I was gaining product knowledge, you might say, in this particular area. In this particular area we were required to know everything about every product, obviously, since we had to test the product before it was packaged and sent to the customer, and it was a learning period, you might say, wherein different categories of products were individually studied by me, and I did most of this at home.

Q And how long did you stay connected with this testing aspect or testing lab?

A I am still doing it.

Q You are still with that -- you are still doing that now?

A Yes, essentially. In other words, through the years I became supervisor of the welding laboratory,

manager ot Technical Services and my present title is director of Technical Services, and has been for a few years.

Q Director of Technical Services?

A Yes.

Q You are a manager of that department, then?

A Yes.

Q What do your present duties involve?

A Essentially, I represent the source for product information. If a customer or a salesman or an employee or any individual, essentially, wants information about our product, I supply it. We, in so doing, become involved in development of products, we become involved in competitive products, we become involved in American Welding Society activities, we become involved in anything and everything to do with welding products. In other words, Technical Services is a service arm of the organization that provides specific knowledge to whoever is asking for this information.

The Quality Control Section still reports to me.

Q When a new product is developed, do you become involved in it at any point?

A Yes, we become involved in the actual testing of it. The R & D people supply samples of potential new products to me, and I assign them to the proper laboratory or the proper technician, and the product is tested and evaluated, and I render opinion on it, since I have done this work myself and still do.

Q Now, is part of your duties to be familiar with competitive products?

A Absolutely.

Q Was that always so or did that evolve at some period of time?

A I would say it was almost always part. It's not -- I shouldn't say it's not official, but rather to me it's just a purely logical thing to do. If I am working on a product or if one of our salesmen asks about a competitor product, I have to be in a position to know something about it.

Q How do you familiarize yourself with these competitive products?

MR. CALIMAFDE: Do you want the question that broad or do you want to focus in on the issue?

MR. SPRUNG: I am talking right now, presently.

A The procedure hasn't changed any. I read

everything I can get my hands on in the way of printed material. I subscribe to several technical magazines. I belong to different technical societies. I acquire technical literature, you might say, and attend meetings and keep abreast that way.

And whenever I see something advertised that I cannot identify, I purchase a sample of it, put it in a lab and test it. And that I have been doing for over 20 years.

Q So you pretty much followed that procedure since you were weaned at Alpine Trading Company?

A Essentially.

Q If you will excuse the expression.

A That's quite all right.

Q Do you keep a file on these competitive products?

A Yes.

Q Do you keep them by competitor product or by type of product?

A Actually, I have a Cardex and cross-indexing system. We use code numbers and, of course, names.

Q Are you familiar with Metco?

A Yes.

Q The Metco products?

A Yes.

Q When did you first become familiar with those products?

A Oh, I believe I studied the wire metallizing system several years ago,and when I became interested in wire metallizing looking at Metco literature and Mogul literature, and, of course, some foreign companies.

Q Could you give us a point of time when you were first familiar with Metco as a company or with their former name which was the Metallizing Engineering Company?

A I would say in the '50's, early '50's.

Q Do you keep a file on the Metco or Metallizing Engineering Company products in your Cardex?

A We keep a file on literature, the brochures that are sent out, whatever they send out in response to one of these reply cards. We keep some of it on file. Some of it we distribute to other areas.

Q Do you keep your Cardex file on these products?

A Well, these are pieces of literature I am talking about now. But we do keep a file on the actual products, too.

Q And you have such a file, a Cardex file or

cross-index file of some type?

A Yes.

MR. CALIMAFDE: He has already said that. I was wondering why you went around it again.

MR. SPRUNG: I want to know if he is indicating the literature that he has gotten.

A No. The Cardex files is not used for literature.

Q It's on the products that have been tested?

A Yes.

Q Has that been produced to us?

A I believe so.

MR. CALIMAFDE: You better wait until Gordon comes in.

MR. SPRUNG: I am going to make a request now that -- I would think that would come under our request of anything concerning Metco's products.

MR. CALIMAFDE: Yes, I would think so, but it may have been produced. I don't know.

Q Do you know if these cards were produced to your attorneys?

A I turned them over to the girl who asked for them. I don't know exactly what she picked up, though. Everything was asked for.

* * *

A We developed the Eutalloy Process, a process wherein powder was injected into an oxi-fuel gas flame and deposited on to the heated work piece.

Q What was your involvement in the development of this Eutalloy Process?

A I worked on the torch itself, several embellishments of the torch, the container in which the powder is packaged, since it is adapted to fit the torch. I worked on some of the powder compositions and, of course, some of the actual applications, quite deeply involved in the Eutalloy Process.

Q Do you have any knowledge of what instigated or motivated -- this was Eutectic at this time in the '50's?

A Yes.

Q -- to proceed and attempt to develop this type of product?

A Well, we had experimented with so-called self-fluxing alloy systems, systems where in a molten condition, a bond would be formed with the base metal without the use of an externally applied flux, and these are compositions that contain, for example, boron, nickel, alloys with boron, so forth and so on.

We had worked with the basic system, the

inconel powder, adding boron to it, conducting little melts in a laboratory, and we had improvised shotting tower. In other words, I don't think there was any moment in time where someone turned around and said, "Let's get into this business," but rather it evolved from a variety of other little products, and this is essentially how new products are developed.

Q At the time that development started on this Eutalloy, weren't you -- when I say you, you personally and Eutectic, familiar with any competitive products? Weren't competitive at that time, but any third party products on the market?

A Yes, we were familiar with products sold by Wall Colmonoy, by Coast Metals, and a few others.

Q And these were nickel-base self-fluxing alloys?

A Some were, yes.

Q Were you familiar at that time with the term the sprayweld process?

A I don't think so. Not as sprayweld. I am not sure if I knew that term at that time.

Q Do you know how Wall Colmonoy marketed their product, under what designation?

A Not really. I know that the Nicrobraze

Series is used primarily as preplaced alloys for furnace brazing. They were rather widely publicized then, and, of course, the American Welding Society brought out a specification on the nickel-brazing alloys about that time.

Q But, basically, these self-fluxing powders would be some way placed on a surface and then heated until their fusing temperature?

A Yes.

Q According to one process they could be placed on the surface by spraying?

A Yes, exactly.

Q Do you know if it was general at that time whether the depositing of the materials and the fusing would be done as a single step or as 2 separate steps?

A Essentially, the processes known at that time were 2 separate steps. The Eutalloy Process at that time was the first process wherein simultaneous application and fusion occurred.

Q Were you personally familiar at that time with these processes?

A Yes.

Q And were you familiar with the fact that when you heated these self-fluxing alloys you can visually

see when they were at about the right temperature?

A Yes.

Q Can you describe that phenomena to us?

A If you apply a self-fluxing material to a higher melting work piece and heated the material it theoretically would melt in stages, assuming it had a plastic range and when the liquidus was exceeded, it would flow over the surface, obviously. There are ways of measuring this and testing the integrity of the bond that you get, so forth and so on.

Q What did the material look like if it was first applied by spraying before it was fused?

MR. CALIMAFDE: Why is this relevant?

MR. SPRUNG: It is highly relevant.

MR. CALIMAFDE: The issue in this case is so well defined.

MR. SPRUNG: I am not going to argue on the issue.

MR. CALIMAFDE: I don't want relevancy to be a criterion of the examination.

Now, I have been refraining from objections to questions which, in my opinion, were totally irrelevant, in the hope that you would eventually focus in on the issues.

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MR. SPRUNG: You are not raising any contentions on difference with Xuper Bond with respect to bonding strength?

MR. CALIMAFDE: Right.

(Recess.)

MR. SPRUNG: As based on plaintiffs' counsel's previous statements, we will not pursue the proprietary bond strength test that plaintiff wished to keep secret.

MR. CALIMAFDE: Okay.

(Time noted: 11:55 a.m.)

(Luncheon recess.)

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A I believe so, yes.

Q Did you evaluate the materials themselves, the Metco 404 and the Metco 450?

A Yes.

MR. GORDON: Which point of time, prior to the development?

MR. SPRUNG: Prior to the development.

A I believe so.

Q Of the Rototec. Prior to starting to work --

A In the normal course of testing competitive products as we evaluated the nickel titanite, exotherm and some other materials from abroad. This business of sampling and testing applies to other countries as well as this country.

Q Do you know if there were any reports available or written in Eutectic concerning the Metco 404 or 450 prior to the starting of work on the development of the Rototec?

A I can't answer that. There may be. There may not be. I cannot report everything I do, and I shouldn't say it that way.

MR. GORDON: The question is: did you have any reports at Eutectic at that time?

* * *

A Well, they are used for similar applications. They are used for similar applications.

Q I would like to refer to Document 50138.

A Okay.

Q And ask you if you are at all familiar with that document.

MR. GORDON: Would you read it first?

A I have never seen it before.

Q You are not familiar with that at all?

A No.

Q It was never discussed with you by Mr. Joseph, Mr. Glaser or Mr. Quaas?

A This memo was not discussed.

Q Going back to 8/11/66, do you recall any discussions or othermemoranda at Eutectic concerning Metco's nickel-aluminide powders?

MR. GORDON: I missed that last question. Before '66?

MR. SPRUNG: At this time, at the date of this memo.

A I don't recall anything specific.

Q Who is Mr. Joseph?

A At this time he was supervisor of R & D at Eutectic.

* * *

case we might recommend modifying the chemistry or making an additive or something.

Q What I am trying to establish is, did you make any recommendations that you recall? As I understand it, you had nothing to do with the composition or particle size of 1-69A or 2-69A.

A No, they were submitted to me for test.

Q And as to the samples designated as 160-1 through 160-6, they were submitted to you for test?

A Yes.

Q And you had no part in recommending their composition?

A Not at that time.

Q When did you first start making suggestions as to particle size, composition or anything else concerning the composition, structure or form of the powders?

A Probably be subsequent to Yourasco submitting the samples. In other words, we routinely test what is sent to us without concerning ourselves too much with the identify of what we are testing, so long as whoever submits the sample tells us what he wants to use as a comparitor, and after this progresses, we hold meetings and discuss it intimately.

Now, I had become aware about that time of Pratt Whitney 1337 specification. It was sent in to us by a salesman, and there was a composition essentially 5 aluminum, 95 nickel, and I believe a particle size distribution, and I made a suggestion to drop the aluminum. Instead of messing with high aluminum percentages, we should drop to this 4½ to 5¼ or 6 per cent range, whatever was called out in the spec.

Q Do you have any idea about the date of this?

A No.

Q It was probably in the middle of 1970?

A I would say so. Yes.

Q Shortly after Yourasco submitted the first samples for testing?

A Possibly, yes.

Q Were you familiar with composition of Metco 450 at that time?

A Probably. I don't recall.

Q Do you recall if it was the same as the Pratt Whitney spec?

A The Pratt Whitney spec calls out a certain product. Now, whether that's the Metco product or not, I didn't know. It does call for an aluminum range, and I believe it calls for a particle size

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am curious to know about it.

MR. GORDON: I am allowing you to answer that question.

A There are powders manufactured by Eutectic that contain exothermic components other than Exotec 29904 and Xuper Bond.

Q When you say "exothermic components," what do you mean?

A A constituent of the powder is exothermic.

Q What I am interested in is any commercial powders marketed today by Eutectic which, when flame-sprayed, will undergo an exothermic reaction or any part of the powders, when flame-sprayed, will undergo an exothermic reaction.

A With that definition the 19910 can be considered as falling under that explanation, definition.

Q Any others?

A So far as others, I am not aware of any.

Q Are you familiar with the present composition of Exotec 29904?

A Yes.

Q Would you tell us what your understanding of that composition is?

A It is essentially 95 per cent nickel and 5

per cent aluminum.

Q What is the form of the powder?

A The form is composite agglomerate.

Q Are you familiar with the manner in which this powder is manufactured at Eutectic?

A No.

Q You have no knowledge of that?

A I have meager knowledge. But no specific knowledge.

Q Would you tell us your meager knowledge for the record?

MR. GORDON: We regard that information as being confidential and choose not to allow the witness to answer that question unless we work out again some protective order.

MR. SPRUNG: You are under the protective order for that.

MR. GORDON: I want to have certain information kept away from employees.

MR. SPRUNG: You just don't approve that this information go to the employees. I can understand your objection if at this point you had consented to someone at Metco seeing this and you said, "Well by consent I don't want to extend to this

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E 1114

# EXCERPTS FROM DEPOSITION OF GLADSTONE H. KAPRALOS

* * *

and successfully sprayed coatings with the Spram torch without fusing.

A Yes.

Q Was any work being done at Eutectic in order to develop or improve or perfect such a process?

A Yes.

Q And this was in the year now, point of time?

A In the Spram area.

Q Can you give me a point of time?

A You see, it started when it turned out that first of all the Spram fusing operation was technique-sensitive, and second of all, when it was found out that there were some uses of the Spram application to spray and not fuse out in the field, and that would have to be 1968, 1969, somewhere around there. You have to understand, this was one of many, many, many projects I was involved with.

I am just trying to place the time as best as I can. I could be off as much as a year.

Q Roughly around 1968 or 1969, to the best of your recollection?

A Yes.

Q I understood that you testified that you previously suggested introducing aluminum into the

Eutalloy process to increase its heat.

A Yes.

Q This was done?

A They told me they had already done that.

Q Who told you they had already done it?

A Well, you see, you have to understand that, as I said, at these meetings we sat and we discussed a number of things, and as I said, Frank Glaser was present, and I don't know as to whether or not you people have looked into Mr. Glaser's background, but Mr. Glaser --

MR. CALIMAFDE: I din't hear a question asking Mr. Glaser's background.

Q I will ask you: If it would be of any help in answering the question would you describe Mr. Glaser's background?

MR. CALIMAFDE: Now I hear a question.

A Mr. Glaser back in 1948 had done original research in nickel-aluminide. In fact, if you look at some of the technical journals and technical publications, you still see literature with an asterisk at the bottom would say "From the work done by Frank W.Glaser."

When we were in these meetings and Glaser was involved, and anything came up on exothermic

reactions, Glaser of course being an expert, always, you know, was fostering an approach along this area, and where I said -- when I remember this, I remember I said, "I got a great idea," because I was new with the company. I was contributing something. I said: add aluminum.

Q Add aluminum specifcally to what?

A To a nickel-base alloy, Eutalloy. They told me, "We have done this."

Q Did they say they did it commercially or experimentally?

A Experimentally.

Q Did they indicate what was the result of this experiment?

A Yes; that it added -- there was an exothermic reaction.

Q Did that give the process any benefit? Did it indicate that?

A They felt that it was an area to be considered. There were other areas of development of Eutalloy, of various powders. It was a good process. They considered that it was more urgent and therefore that was something that was going to be investigated later.

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Q Were you able in your search of the files to find any documents that reflected this?

A This was a discussion. As far as the work was concerned?

Q Yes.

A I don't think so. That's going back. I don't think so, But I might be wrong. It might be there in the documents.

Q Would you check?

A Yes.

Q Now, was it discussed what form they were to add to the aluminum?

A Yes. They mechanically mixed it.

Q What was the idea in adding the aluminum?

A To increase the heat output.

Q In what way would it increase the heat output?

A By the formation of an exothermic reaction.

Q With what?

A With the nickel-base alloy.

Q In other words, you felt that the aluminum would react with the nickel in -- how did you conceive that such a reaction would take place? Do you know the chemistry of it?

A I just finished telling you that Glaser was there, and he was an expert in developing nickel-aluminide from the beginning, from 1948. So I would assume that he might just -- since he did the basic work on it -- might conceive that nickel and aluminum mixed would cause an exothermic reaction and increase the heat.

Q I must have misunderstood. I thought that you had said that this was your original suggestion.

A Yes. I said I suggested it, and they told me they had already done it.

Q As for your suggestion, what was your idea how the aluminum would react?

A I felt that it would react in the same way that with my experience with the thermit material.

Q Do you know how a thermit material would react?

A Yes.

Q How does a thermit --

A The aluminum reduces the oxide. Thermit is iron oxide and aluminum, and aluminum reduces the oxide, and there is heat given off.

Q You are fairly certain of that?

A I am pretty sure.

Q And that's what you had in mind?

A I had in mind, yes.

Q What was the purpose of this heat?

A As I said to you, one of the problems or the elimination that we had in the output of the Eutalloy torch was the amount of heat being put out.

Q In other words, this was to make the torch flame hotter?

A Yes; this was to make the deposit hotter, produce more heat, more heat energy.

Q In a process in which the coating as it was being deposited was being fused by the torch flame; right?

A Yes.

Q Was anything pursued on this suggestion after it was made?

A No, They told me -- once I made the suggestion, I realized that first they had tried it, and second I realized that there was a lot of expertise there in this area. But it was a project that, as I said, was on the back burner, so to speak, because there were other projects that were considered more important.

Q Was a project on the back burner, in other

words, they were considering resuming work on it?

A It was an idea that was -- that had merit to follow through, but there were other areas that were worked on.

I think if you look at that period of time there were a number of Eutalloy alloys developed for patents -- where patents were received. There was sample work to keep. They were doing research in an area where they felt it was something that had more commercial value.

Q You said this suggestion was fairly soon after you arrived at Eutectic?

A Yes.

Q Would the date be around '65?

A Yes. They told me they had already done it.

Q Had they done it successfully?

A They had gotten heat, but they also had gotten some additional inclusions, as they felt that it was something to work on.

Q Had they made a satisfactory coating by doing this at this time?

A I think the word "satisfactory" has, you know, it has very broad meaning.

From a Eutectic point of view, something

satisfactory is something that is extremely easy to operate and not technique-sensitive and so on. And I think they had gotten a workable coating. But they were not satisfied with it.

Q What's the basis for your statement --

A Because they told me.

Q Who told you?

A Quaas.

Q You said they had tried it, they got a good coating, but they are not working on it anymore?

A They said they tried it, they got a deposit that's workable, and it is something to consider for a future product. But that there were other products that were more important at the time that were being worked on.

Q So they did nothing further on it?

A No.

Q During your period of time at Eutectic was that project ever picked up?

A What happened was this: --

Q I didn't hear your answer.

A Yes. What happened was this: In June, shortly thereafter, I think it was maybe 1966, work was begun on what we call the Eutallite products, and this involved

the agglomeration of alloys, products and fluxes and like materials on the surface of a base powder. And this was a very interesting project. And in doing that work, I think it occurred to me and Quaas quite easily that we tried a whole slew of systems. I think -- I don't know. That's where I learned my alloys and how many alloys there were. Because we tried a whole agglomeration of a whole slew of systems. It was of course very easy, and this thing worked quite well.

Q What?

A The Eutallite process, the agglomeration of fluxes and/or metallic powders on top of a powder core.

Q As I understood -- and correct me if I was wrong -- that your Eutallite processed involved the coating of fluxing materials on the surface of your alloy powders?

A No. It was metals as well as fluxing materials as well as cermet.

Q Do you have any documents reflecting --

A The research that was done?

Q Yes; on the Eutallite.

A Yes.

Q Could you produce those?

A I don't know.

Q Let me ask you this: Is your testimony -- you were testifying as to the addition of aluminum to Eutalloy alloys, and I said what further work had you done on this, and now you are talking about --

A I am explaining to you that with the development of the Eutallite process where they agglomerate.

Q In your Eutallite process and Eutallite system did youagglomerate aluminum?

A Yes, definitely.

Q That's why I would like to see those documents. "and it became very obvious."

You keep saying it became very obvious. You are talking to yourself?

A I am answering you.

Q When you say it became obvious, are you talking about to you?

A Yes. We realized of course it was very simple to consider agglomerating aluminum and nickel. You are getting the same exothermic reaction.

Q That was your idea?

A I wouldn't even consider it worthy of an

idea. It was an obvious fact. It was very simple.

Q This was in 19 --

A '67. Let '66, '67, where this whole Eutallite program came into effect.

Q Do you have any experimental data showing the agglomeration of aluminum on nickel?

A I don't know. I would have to look.

Q Isn't the Rototec process the agglomeration of aluminum on nickel?

A The process itself?

Q No; I mean the product, the consumerable.

A Which?

Q Your initial bonding material.

A Yes.

Q Isn't this the agglomeration of aluminum on nickel?

A Yes.

Q So wouldn't in the request for documents --

A Okay. You see, I misunderstood you. Once we had the Eutallite process it was pretty obvious to everyone that --

Q When you say "everyone" --

A To everyone at Eutectic's research department, Quass, myself, Glaser, Enderick. -- that it would be

MR. SPRUNG: You will have an opportunity to cross-examine.

Q You said it was so obvious.

A It was obvious that if you take certain materials including nickel, aluminum, and put them together, you apply heat and you get an exothermic reaction. That's obvious. If you ask me what causes the exothermic reaction --

Q Why was it obvious, because you knew of Glaser's work of intermetallic--

A No, because, number one, I learned it in metallurgy in college.

Q Let's get back. Do you recall the text--

A You didn't let me answer my question why it was obvious.

Q I want to get one at a time.

A You said why was it obvious. I have to answer you there were a number of reasons why it was obvious.

Q Yes.

A One would be because it was known, it was known in the public knowledge. Number two, it was --

Q I want to find out where in the --

A Let me finish my answer. Number two, it was

obvious because we discussed it earlier that Glaser had done it. Number three, it was obvious because I had suggested thinking that the addition of aluminum would produce heat earlier, thinking of the thermit experience, and they had explained to me that yes, it does produce heat, is an exothermic reaction, but it is not the same kind of reaction as it is with thermit.

Q Who explained it to you?

A Quaas, Glaser, Broderick.

Q They said it wasn't the same?

A It is not the same.

Q Did they explain what reaction it was that it produced?

A I don't recall.

Q Did they mention intermetallic?

A They mentioned what it was at the time. I am not versed well enough in it to explain it to you.

I am not an expert in it.

Q You do recall they said it was different than a thermit?

A Yes. Those were the reasons why it was obvious. You can go to each one if you like.

Q Can you designate what materials in the public

et

domain, publication you are referring to?

A I remember in college reading about that stuff in the library. Come on now.

Q You do?

A Of course.

Q Do you recall the textbooks, in other words, that nickel as a metal and aluminum as a metal would interreact exothermically?

A Of course.

Q And that's the type of exothermic reaction you were thinking of?

A Of course.

Q At least your concept of doing this was to increase the heat in the Eutalloy process?

A Of doing what, when, where, how?

Q You were just saying that once you developed your Eutallite process it became obvious that you should take aluminum and agglomerate it with nickel, that was for increasing the heat.

A That was the original area that I got involved in. Okay.

Now, once it became obvious, it wasn't really a project that was ever a project of great importance, but it was something of interest. It was

* * *

A (Continuing) Yes.

Q You were solely in charge of its development?

A There was no charge. I assumed it. As I was going to mention to you before, Mr. Yourasco, who was our then metallurgist, and I were riding in the car one day, returning from a trip we had taken.

Q About what time?

A '69, '70. Either late '69 or early '70. And I told Yourasco, "Why don't you take some nickel powder," I think I told him, because he had nickel powder handy, "and take some aluminum and use the Eutallite process and make some agglomerate particles similar to Eutallite of aluminum and nickel.

Q The Eutallite process, has that been described in any publication?

A Possibly. I am not sure. No, the product process would not. The Eutallite series of products were just in the Eutalloy process.

Q Did Mr. Joseph have anything to do with Eutallite?

A Yes.

Q Was he the primary developer of that?

A Yes.

* * *

not the only product I have done that with, incidentally. There were other products that I did the same thing with.

Q This was "your baby" so to speak?

A It was interesting to me.

Q Why was it interesting to you?

A For a number of reasons. Number one, it was one of the things I had gotten into when I had gotten in the company. Number two, I always had the feeling that this might be of interest to Eutectic, and number three, because Glaser was involved with nickel-aluminide earlier. He was the boss.

Q When you say "nickel-aluminide", you mean a compound between nickel and aluminum?

A Yes. He was involved in nickel-aluminide research when nickel-aluminide first came into being, I guess.

Q What did you expect when you told Yourasco to make this?

A I expected him to come up with a powder that was manufactured similar to the Eutallite powder, exactly the same as the Eutallite powder and with some modifications I expected to have a powder that we would take and test through various torches.

Q How did you expect this to perform? What did you expect of it, anything special?

A To perform?

Q Yes.

A Yes. I expected that there would be an exothermic reaction connected with it, and I expected that I would get, because of the exothermic reaction, which means a high heat output, I would get some kind of a bonding effect on a base metal that might have some interest.

Q What was the basis for your belief that you would get some sort of a bonding effect on the base metal?

A Because that's the basis of metal. You have high heat and you got bonding.

Q Do you know of any commercially available or described materials in which sprayed particles bond because of an exothermic reaction prior to this time?

MR. CALIMAFDE: Does he know now or did he know?

MR. SPRUNG: I am talking about the time that he made the suggestion to Yourasco and he thought he was making this because he expected it to bond.

A You see, I can't really pin down --

* * *

Q I said which would have an exothermic reaction.

A I interpreted that to mean that something that would have sufficient heat to bond, that one doesn't connect with the other. I am not telling you that I knew it would bond because of something else that was being sold.

Q What I am trying to establish is: Was there anything other than the information you got which emanated from Metco which would indicate to you because of this exothermic reaction the material would bond?

A I had knowledge all along. The first thing as I got involved was when I got into Eutectic I was interested in that from then. When you are interested in a subject, you think about it from time to time.

Q What I am trying to establish is what told you or what was the basis of your knowledge that a material which would exothermically react would bond to a surface.

A If I take a coin and I let it go, what would happen to that coin? It would drop. It is common knowledge.

I think that anybody with any more technology like that would have known its involved in

that kind of an atmosphere.

Q In other words, it was your understanding that it was well known in the art that if you heated a particle hot enough it would bond to the surface?

A Of course.

Q Do you know of any in the flame-spray field, any materials that would perform other than molybdenum in this manner?

A I think that if you heat a number of different alloys you just spray them and they would bond.

Q What is the basis of your thought?

A Because if you have a high enough heat you are going to get a bonding.

Q Have you ever tried it?

A I said before I tried it in connection with some nickel-base alloys, and I sprayed it, and it bonded.

Q What did you spray?

A The nickel-base alloys.

Q What made them hotter? I thought you said you sprayed them in the normal manner.

A Yes. If you heat them beyond a certain temperature, they are going to bond.

* * *

Metco's product or its impact on the field that caused Eutectic to try --

A I didn't know too much about Metco's impact on the field. I knew the product and I knew the patent, yes. I didn't know what their market was. In fact, I will tell you, I don't know what their market is today.

Q After this first initial experiment that Mr. Yourasco made of this material, what did you do with it?

A John Broderick tested it.

Q How did he test it?

A Sprayed it.

Q And what percentages did you tell Mr. Yourasco to use of the nickel and aluminum?

A I think I went 40 on down, 40 per cent of each on down.

Q Did he follow those instructions?

A I think somewhat. Not 100 per cent.

Q Did he write a report on the first material he made?

A No. He submitted them to Broderick. All he said is, "Here is this material. Kapralos wants them tested."

* * *

A Yes. I think it was in the material I gave Mr. Gordon.

(Recess.)

BY MR. SPRUNG:

Q I hand you a document which I will ask the reporter to mark Defendant's Exhibit 2, and ask you if you can identify that? The base number is not on the one I have. But it is No. 50100.

A 50100. This seems to be the initial batch of composite agglomerated powder that Mr. Yourasco prepared, the first six samples.

(Document bearing Bates number 50100 marked as Defendant's Exhibit 2 for identification.)

Q Did you give him instructions, suggestions on how to prepare them?

A Only to use the Eutallite process. That meant a specific finale and so on.

Q Did you indicate what was to be the base material and what was to be put on it?

A No. I had told him nickel and aluminum powder. I see here he used some alloy, nickel alloy powder, he used some pure nickel powder, he used some nickel oxide. So he used his imagination. The analyses are below.

Q Then you did not suggest the proportions of nickel and aluminum?

A I told him 40 per cent on down, but I see he used his own.

Q Let's start in the 60-1.

A 160-1.

Q Is that just a code sample number?

A That was his. He made it up.

Q It says nickel base powder, 400. What does that mean?

A I think the 400 is the number of grams.

Q And aluminum, 72, that's the number of grams?

A It could be a code. I am not sure. It could be either one. I see below he has got the final chemical of the deposit. It could be the amount of weight or it could be a code. I am not sure.

No, it is a code.

Q What is a code—

A I can't answer that. I am guessing. I can't. It could be the weight. It could be a code.

Q The final chemistry exclusive of binder, does that indicate the proportions of nickel to aluminum?

A Yes.

Q Is that weight per cent?

A Yes.

Q I refer to specimen sample No. 160-1, that had 82.4 per cent for nickel and 15.20 aluminum; is that correct?

A And .60 silicon and .76 boron.

Q And sample 160.2 has about 64 to 15 per cent of nickel to aluminum; is that correct?

MR. CALIMAFDE: Are you asking whether this exhibit has these figures? It speaks for itself.

MR. SPRUNG: I am trying to interpret it and get it straight for the record. I am trying to say does that indicate that that is the percentage of the nickel to aluminum in the powder.

THE WITNESS: Yes.

Q In the final chemistry, that's what I am trying to establish.

What's the highest percentage of aluminum that they are using in that? Is that 20.8 per cent in sample number 160.5?

A Yes, 20.8 per cent aluminum, 16.8 per cent oxygen. What he was really using, if you look on top, he was -- must have been using some kind of an oxide.

Q Nickel oxide.

What I am trying to establish is that the highest amount of aluminum in these specimens is about 20 per cent; is that right?

A Yes. In the one he used nickel oxide, yes.

Q But you indicated that you had suggested that he try from 40 per cent down?

A Yes. I recall that.

Q But he didn't follow your suggestion.

A No.

Q Do you know what made him pick these figures?

A He was the technical man. I don't know whatever the reason was.

Q Do you know if he had any knowledge of Metco's products at that time, George Yourasco?

A I don't know specifically if he had knowledge, but he is a good metallurgist.

Q You didn't discuss with him the fact that Metco had such a product on the market?

A No.

Q You didn't show him the patent which described how to make the product?

A No.

Q And then these were -- I see at the bottom it says "Initial bond test." What does that mean?

A This came from Yourasco's records, so I think I can take a reasonably good guess and say that this was his initial spread of what he produced; then Broderick tested it, these samples, and came back and reported the initial bond test of the samples. 1, 2, 3, 4 were okay, and two were not good.

Q Do you know how those bond tests were made?

A No. I have to have Broderick testify on that?

Q And you would, to the best of your ability, date this document back about what period of time?

A Again, I have to say late '69, early '70.

Q To the best of your knowledge this is the first time that Eutectic made such a powder as an agglomerate or composite of nickel and aluminum?

A Unless --

Q I am talking to your own knowledge.

A Yes. Yes, unless, as I said, I can be corrected by somebody else.

Q To your present knowledge, do you know of anything earlier?

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sprayer to which the patents were directed?

A No. I don't think so. I think I just asked if it was valid or not. I may have given some background as to some of the things that we had done that I testified before, but I don't recall. I just don't recall.

MR. GORDON: Off the record.

(Discussion held off the record.)

BY MR. SPRUNG:

Q I refer now to Defendant's Exhibit 2. After the work that's depicted in this report was completed, what was the next step or instructions that you gave on this development?

A I asked that they do sufficient development work to come up with a product.

Q You say "they". Who?

A Yourasco in terms of development work and Broderick in terms of testing.

Q You gave instructions to Yourasco and Broderick and said, Come up with a commercial product?

A Yes.

Q Did you give them any indications how they should go about it?

A No.

Q Do you personally know what they did after receiving these instructions?

A There was a series of tests, Yourasco was making product, and Broderick was testing it. Very shortly after their, when Broderi k had several versions that he felt were the -- were good, we brought in the marketing people, and we gave the product to one of the people that -- one of the salesmen, or several of the salesmen, to go out and market what we called market-test the product, which was to show it and use it in various applications in with the Spram gun.

Q We established an approximate date for that, did we not?

A Yes.

Q When was that?

A Late '69, early '70.

Q Just at the turn of '69, '70. And you said you told Yourasco and Broderick to develop it into a commercial product. Did you get authorization from anyone at Eutectic to do that?

A No, I had that authority.

Q How soon thereafter did they make this commercial product?

A Very short time, a few months.

Q A few months?

A Yes.

Q Did they work with intensity during --

A No, it was a matter of Yourasco. This was something that he was doing in between other work. His main responsibility was in producing powders, the various powders that Eutectic markets, in running that division.

Q He was just doing this as incidental work?

A He got his $200.

Q It wasn't considered a very important program?

A It was -- to me it was an interesting project, because I was involved. You have to understand. As an engineer and attorney, I was really interested in coming up with the project -- with products that would be good marketably, and to me it was an interesting product, and there was some development work, and until we gave it out to the Spram people to go out and test it in the field.

Q When you say "to the Spram people" --

A We gave it to a couple of salesman that were trained in the Spram process.

Q They were Eutectic sales people?

A Yes. They were experienced in our Spram process that we discussed.

Q What was the first date you gave it?

A I don't recall. Within a few months.

Q How much man-hour time was spent in your estimation from the time of the first report until you got the initial product out for testing?

A I can't guess. There was enough, though -- each test takes a period of time because I remember there was one point where Broderick had commented to me that these tests were taking up some of these people's time for -- that he had for other products.

Q But Broderick was testing.

A Yourasco was producing various types of combinations.

Q Were you spraying the materials you produced?

A Yes.

Q Were you testing?

A That's what I mean, spraying and testing.

Q What kind of test did they do?

A I am going to have to let Broderick-- you see, what he did, once Broderick discovered that this

was a nickel-aluminum combination, he of course picked up a sample of the Metco, one of the Metco products, and was testing performence of these products up against the Metco product, and this is standard with us. When we test our products, we will test them up against a competitor product, if such exists.

Q When did you start buying and testing against the Metco product?

A I would assume once Broderick knew what was involved, then it would be natural for him to do that. So I would assume right away.

Q I notice the only testing that is indicated on Defendant's Exhibit 2 is an initial bond test.

A That's probably the first test that Broderick made.

Q Do you normally on your products make the bond test?

A Yes. It is very important. Not only in the Eutalloy, which is a flame-spray process, but also all kinds of processes where you are talking about electrodes, welding electrodes.

Q So you were competent in making bond tests, Broderick was?

A Yes.

Q It was standard procedure for you?

A I can't call it standard procedure. I was not familiar with the kinds of testing that Mr. Broderick did. That was an area that I didn't get involved in. All I know is that Broderick tested material, and Broderick is a pretty competent man, people under Broderick's supervision.

Q When you had Yourasco first make the samples, did you suggest how that should be sprayed?

A No. I told Broderick to spray it. Here is another thing that's pretty normal. Whenever Broderick sees a material, or anyone for that matter in the research area sees a material advertised, that might be sprayable, that might be used in some other way, he will order and then they will try it in many different ways. One material would be tried in many different ways. I am sure he tested it through a number of different systems.

Q He didn't report in depth to you on that?

A I was only interested in receiving from him an idea. I wasn't interested in detail. I wasn't-- I was interested that it was a good product that we can go from there and release it commercially.

Q Did you get such an indication after the

* * *

MR. GORDON: Knowledge meaning what, the existence of the product?

Q Did you know that Metco had a 450 powder?

A I am not sure. I am not sure, as I said, I didn't know too much about the Metco products. Now, if I had-- when you say knowledge, I assume whether I heard it.

Q Did you know or do you have a recollection at the time you asked Mr. Yourasco to make these samples which were ultimately made and reported in Exhibit 2 and for which you offered him the bonus, that Metco had a product designated 450?

A No, I don't think so.

Q You didn't know?

A No.

Q So then you didn't know of Metco's 450 product in 1968?

A No. You see; I have to say something. When I became employed by Eutectic, I was new to the welding industry, so over the next three, four years, I may have heard names, but when you say knowledge, I have to assume and interpret knowledge as a specific recollection, and not something that I might have heard somebody mention which didn't mean anything

to me and might-- something which I might have seen in a magazine, in a welding magazine or something. But specific knowledge of knowing exactly what something means, and on the basis of that definition, I have to say no. I didn't know the word 450 or what the product was. Now, I am not saying-- I am not saying that somebody may not have mentioned it to me, but no specific knowledge and recollection.

Q As I understand that while you received knowledge of the Metco patent 3,322,515 in '68 and had a validity study made by outside counsel, you never inquired as to whether Metco had a commercial product which they offered under that patent?

A When I ran into the Metco patent which was a little earlier than that report, first thing I did was I ordered a D and B.

MR. GORDON: The question is whether or not you knew about a product they had on the market.

A Whether they had a specific commercial product at that time that covered the patent?

MR. GORDON: Listen to the question.

Q Yes. In other words, whether they were offering anything on the market as was described in their patent.

MR. GORDON: The question is at that time did he make a study to find out if they had such product on the market?

MR. SPRUNG: An inquiry.

A Not a specific study, no.

Q Did you make any inquiries?

A When the patent came up, I may have talked to Quaas and Broderick, and Quaas and Broderick are very knowledgeable in terms of what goes on in the industry, and they may have mentioned it.

MR. GORDON: Do you have any recollection of any such conversation?

THE WITNESS: No. I don't specifically recollect. But I don't want to say it never happened.

Q But you had no real interest to find out or analyze-- find out and then analyze any commercial products made under the patent?

A No. Not by a competitor, no. We were interested-- I was interested in what I was going to do.

Q Didn't it occur to you if there was such a product on the market it would be the

easiest way of finding out how it worked and how it was made?

A No. I don't think that way.

Q So your earliest recollection at this time of the Metco 450 product was about the time you wrote this letter?

A No. My earliest recollection of the fact that Metco had products of this type was really when Broderick started to test what we had developed up against those products.

Q Who brought those products to Broderick's attention?

A As I said before, Broderick is very knowledgeable in the welding field.

MR. GORDON: The question assumes that somebody brought it to Broderick.

Q Do you know if anyone brought it to Broderick's attention?

A Broderick is the one who brings products to other people's attention.

Q So you have no personal knowledge of anyone bringing this to Broderick's attention or writing any memorandum on it in Eutectic?

A No.

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A Yes, that's right.

(Luncheon recess taken at 11:55 A.M.)

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AFTERNOON SESSION

1:00 P.M.

G L A D S T O N E K A P R A L O S , resumed.

EXAMINATION BY MR. SPRUNG:

Q Mr. Kapralos, do you still maintain an office at Eutectic?

A Yes.

Q Do you have a secretary there?

A She is referred to as my secretary. She worked for someone else who's left, but once they rehire a new lawyer, she is going to be his secretary. But right now no.

Q But right now you have an office and a secretary at Eutectic?

A Yes.

Q Mr. Kapralos, when you developed the composite nickel-aluminum powder starting with Defendant's Exhibit 2, at any point thereafter did you consider filing a patent application covering that development?

A No.

HCpa64 Kapralos

Q You never did?

A No.

Q Did your knowledge of the Metco patent have any influence on you?

A No.

Q What reason did you not consider filing a patent application?

A I knew that the entire system was well-known. I didn't think it was patentable.

Q But you did subsequently add another component to it and file a patent application?

A Yes.

Q That component changed the basic nature of the material?

A I don't understand what you mean by that.

Q In other words, the component you added, did it change the material in any way?

MR. GORDON: Change which material?

MR. SPRUNG: The nickel-aluminum combination.

A It changed the structure, it changed the operation, it changed the result, as far as I was concerned.

MR. GORDON: Are you saying-- did it

Q Did you evaluate those test reports?

A I didn't evaluate anything. Broderick is the one that evaluates.

Q I am trying to determine the basis for your statement that the powder was better than Metco.

A Our technical people told us.

Q Are you familiar with any Broderick reports which indicate it wasn't as good as the Metco powder?

A No.

Q But you have read the reports?

A I have seen reports, yes, and I have glanced through them. But not evaluate them.

Q In other words, your opinion that it was better than Metco is just what Broderick told you?

A Yes.

Q Was it a coincidence that the powder was equivalent in every respect to the Metco 450 product?

A What do you mean by "coincidence"?

Q Was the Metco 450 product copied or did it just happen that it came out when you finished the development, it came out to be the equivalent of

the 450 in every respect?

A I think that as I said , somewhere along the line Broderick started to test against the 450 and he found out the product we had developed was as good as and better.

Q Now, do you know if the Metco 450 powder at any time prior to this July, 1970 date was analyzed at Eutectic for Eutectic?

A I have no personal knowledge of that.

Q You never saw any reports of analysis of the Metco product?

A No.

Q Did you know how much aluminum was in the Metco product?

A I know now.

MR. GORDON: As of July, 1970.

THE WITNESS: I acquired that knowledge. I can't say when.

Q Did you know the composition of B.N.9048 as of July 10, 1970?

A No, not the specific composition, no.

Q Did you know how much aluminum and how much nickel it had and what the particle size was?

A No.

* * *

couple of years.

Q Do you have any literature references that you can think of that confirm that?

A It is personal field trips.

Q Would these field trips involve Eutectic products mainly?

A It involved what I was able to see in the field and some of these shops they had a variety of products and a variety of processes.

Q Metco's nickel aluminide materials, that you are familiar with, did you know at that time whether they were intended to be applied by a cold process, hot process or both?

A At this time, 3/23/70?

Q Yes.

A No.

Q You had no knowledge?

A No.

Q As I understood your previous testimony, while you had uncovered the Metco patent, and that is number 322515, and had obtained an opinion, Exhibit 1, on validity, you had not read the text of that patent, is that correct?

A That's right, I had not read the text of that

patent in complete detail. I read the claims in detail.

Q When you say in complete detail, have you read the text at all?

A I think I read the very beginning, and then I skipped through to the claims.

Q When you say the very beginning, the introduction?

A Yes.

Q I hand you now a copy of patent 3322515. Can you tell me about how far in the introduction you had read?

A Up to about line 40.

Q Did you recall any definition of flame spraying in that patent at the time you read it?

A I frankly related flame spraying to our process.

Q You made no notation that the patent related to what has been defined in this testimony as a cold process?

A I didn't even have the slightest idea what a cold process was.

Q At that time?

A That's right.

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this context?

A My understanding is that materials upon being heated will give off heat.

Q And those are exothermic bonding materials?

A Yes.

Q What does the term bonding mean to you?

A Bonding to me means joining, alloying, gluing.

Q It says "Prior to the advent of these exothermic bonding materials." Do you know what materials you were referring to as the exothermic bonding materials, by name or designation? Wereyou referring to your ExoTec material?

A Yes.

Q To Metco 405, and Metco 450?

A No. To our ExoTec.

Q That's all?

A Right.

Q So prior to your ExoTec, molybdenum wire sprayed via wire gun was the only material that oculd be utilized as a base bond undercoat for subsequent metalized surfaces. Is that what you meant?

A Yes, but for the most part this is a sales tool and marketing tool prepared by several people.

Q I am asking what it means.

A What I am telling you is, the meaning of that has

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# EXCERPTS FROM DEPOSITION OF BERNON A. COOK

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some form of preparation is always necessary.

Q What do you mean by mechanical?

A The interlocking of minute particles into a prepared surface. There are several methods employed in that. Number one, a blasting method, where a substance such as a steel grit or a carborendum or sharp silicon sand is used to clean and scarify the surface to provide minute crevices for holding on the sprayed material. Another method is to machine the surface and turn a rough thread setting the tool with a negative rake, somewhat below the axis, and to tear the sides of the thread and this forms crevices in which sprayed metal is locked.

Another method is to use an exothermic material, similar to, say, a molybdenum or the material that is in question here, and form a locking surface onto the surface before it is sprayed.

Another method is to use a bonding material which is with an electric transformer type unit, a nickel type electrode, to create a crevice on the surface or cavities that will hold on the sprayed material.

Q How long have you been involved in the flame spraying metallizing field?

A Most of the time it has been in existence in the United States, since 1934.

Q And in your experience is the bonding of the spray material to the surface being flame sprayed a problem?

MR. GORDON: The question isn't clear to me.

Q In your experience has it been a problem to secure bonding of the flame sprayed material to the surface being sprayed?

A There have always been methods for doing it.

Q If you just spray on a clean sprayed surface, will the spray material stick?

A The only exception to that is with the arc spray system.

Q Normally when you spray a machine shaft that's merely cleaned and emeried, the spray metal won't stick, is that right?

A That is correct.

Q Was your company back, many, many years ago that first proposed the rough threading and undercutting of steel shafts in order to secure a bond to the material?

A That is right.

Q And then to grit blast it so you made

undercuts so that the spray would mechanically bond?

A Grit blasting is an alternate method. It is not necessary to use together.

Q I am saying two separate methods, mechanically undercutting either by screw threading and turning the threads or by grit blasting.

A Yes.

Q A mechanical undercutting of the surface?

A Yes.

Q And that was developed about when, according to your understanding?

A The first development was approximately 1934, but it has been modified through experience over the years. Almost since the beginning of the industry.

Q And that was the standard used in the industry in order to prepare a surface for flame spraying for how many years, would you say?

A It's still used today.

Q For how many years, would you say?

MR. GORDON: From '34 to '73.

A Almost 39 years, 38, 39 years, with modifications of it, different procedures.

Q All some mechanical cutting into the surface in some way, is that correct?

A That is right.

Q Are you familiar with a process that was developed a little later, of electrically welding little spots on the surface?

A Yes, I just described it.

Q When about did that come into effect?

A You know, I just can't answer that. In the 1940's. I don' t recall.

Q Sometime in the 1940's.

A Yes. I would say approximately, don't hold me to it, about 1944.

Q When that method was developed your company started using it and promoting it, for bonding?

A That is right.

Q If I say to you a self-bonding flame spray material, do you know what I mean?

A Yes, I do.

Q What is a self-bonding flame spray material?

A Well, molybdenum is self-bonding in general a material--

Q Which will adhere to the surface?

A Yes, to a clean surface.

Q And once a flame spray material of a self-bonding material, subsequent coats will stick to it because the flame spray material is in itself rough, is that correct?

A That is correct. In other words, you get a sprayed surface, and you can build, with certain limitations, other material on top of it.

There are limits on that. It isn't universal.

Q That I understand. Mr. Cook, up to and through the time of this mechanical surface preparation, and the time that the electrode preparation of the surfaces were developed, do you know if there were any self-bonding materials known or used in the industry?

A The first self-bonding material that I personally knew of came in the 1940's.

Q Which was that?

A Molybdenum.

Q Late 40's?

A I would have to refer to-- I would say possibly the mid-40's.

Q And that was the first self-bonding material, that from your experience, you knew existed

in the flame spray field?

A Yes, it had been successfully applied.

Q Do you know who Metco is?

A Yes.

Q Do you know if they used such a process under the name of Fuse Bond?

A Yes, they did. What they are marketing it now I don't know.

Q But you knew it at the time it came out?

A That's right. We both had it.

Q To your knowledge, who was the first one to come out with the use of molybdenum, as a self-bonding material?

A I am not sure that I can answer that. I believe it was-- don't hold me to it-- it was General Electric.

Q You had no knowledge of Metcocoming out with moly bond, a spray bond?

A Yes, I had knowledge of it but I don't know whether I had-- we had knowledge at the particular time they came out of it. Everybody was selling molybdenum and always had.

Q After it came, sometime in the 40's--

A Yes, and we are selling it today.

Q At the time that molybdenum came out on the market and started being used as a bonding material, was it commercially successful, to your knowledge?

A Yes.

MR. GORDON: How do you define commercially successful?

Q Was it very widely used and promoted in the flame spray field?

A Quite widely used, yes, and quite broadly used, yes.

Q Did you know of any other material which was self-bonding, other than molybdenum?

MR. GORDON: At which time?

MR. SPRUNG: At any time.

A Not in wide use that I know of.

Q Even as of today? I am talking up to the present.

A Up to the present?

Q Yes.

A Well, the material in question is in use.

Q That is a self-bonding material?

A It's a self-bonding material.

Q What is your understanding of that material, what it is?

A Well , it provides a bond or a build up job, very similar to what the molybdenum is. There is an alternate to it, or a supplement to it.

Q Do you know, Mr. Cook, what the material is, metallurgically or physically?

A Not particularly because we don't manufacture materials.

Q Do you have any knowledge that it contains nickel and aluminum which exothermically react?

A Yes. This is what we read and this is what we have reason to believe.

Q When did you have first knowledge of such material coming on the market as a bonding material, self-bonding material?

A From an article that was mailed, that appeared in Iron Age, and was mailed to me from Sweden.

Q About what period of time, do you have any idea, to the best of your recollection?

A I think about-- I have to refer to that, but it's about 1966, or '67.

Q I hand you a document which I will ask the reporter to mark Defendant's 83 and 83-A.

(Document marked Defendant's Exhibit 83 and 83-A for identification.)

Q Is 83-A the article you are referring to?

A It is, 1963.

Q Up to that time, had you known of any self-bonding materials available or used, in flame spraying, other than molybdenum?

A No, I don't think so.

Q After this article, and subsequent to it, did you become familiar with this type of material as a self-bonding material, the material of the general type referred to here?

A I don't know if we ever came too familiar with the material as a structure because we don't build materials. We attempted or I instructed our people to investigate to see what might be available on the market, and we had some information from Sherritt Gordon in Canada, and we had a number of other reports but we were not marketing or doing anything with it, nor had we been able to determine any known user that was employing the material.

Q This is in '63?

A That's right.

Q When did you first become familiar with any known user who was employing the material, if you did?

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MR. SPRUNG: He is an expert in this field.

MR. GORDON: He sells products.

A We are machine builders and marketers of machines, and I am not personally in the field. We know of places where it is used but to what extent I can't--

Q Does your company recommend this type of material?

A We have.

Q Do you know any applications where it can be used, where molybdenum cannot be used?

A Only from certain reports where they are going to build up,like in a jet engine blade or housing of a blade, where it is softer than molybdenum but from a bonding standpoint we cannot determine very much difference between the union between a wire molybdenum or a powder of this type as far as bond strength or quality is concerned but it is a convenience, too, because in other words, if you are running a powder and you are going to build a powder overlay on it it's one operation. Molybdenum powder in spray form is completely unsatisfactory.

Q Do you know prior to the advent of this type

of material as depicted in Defendant's Exhibit 83 and 83-A, whether there was any self-bonding material available for the powder flame spray process?

A I believe Wall-Colmonoy had a powder. I am not too well acquainted with it.

Q Do you know of any self-bond flame spraying powder that was used in the flame spray field prior to the advent of this powder?

A Personally I don't know, but I would have to refer to our marketing people.

Q Has your company, to your knowledge, at any time marketed or promoted any self-bonding powder other than this type of material?

A No, sir.

Q Do you know, from your own knowledge, whether anyone else did?

A Not in our company.

Q Any other company?

A I wouldn't know. Let me make my position clear. I am not involved with our technical end of it altogether. My duties are administrative. Many things can happen which I don't personally have knowledge of, so I will have to--

Q That's understood, Mr. Cook.

* * *

A I recognize this as being good.

MR. GORDON: Isthis being offered as an exhibit?

MR. GORDON: No. It's a publication.

Q At present is your company manufacturing or selling any exothermic self-bonding flame spraying materials?

MR. GORDON: Why don't you ask that in two parts, manufacturing and then selling?

Q Are you marketing any--

A We are not manufacturing. We never have manufactured, and don't manufacture materials of any nature whatsoever, and never in the history of our company have we ever made one powder material. We buy materials from the open market. We are selling molybdenum, and we have this material that's in question, but we do not market the form of it in the wire.

Q What exothermic self-bonding material are you marketing?

A Eutectic powder.

Q Under what designation do you market it?

A We have a number 55, we label it. All materials bear our label.

Q Under M-55?

* * *

A That is right.

Q Does your company market any other self-bonding materials other than the molybdenum and this Mogul 55?

A Hard facing powder alloys, and that's all.

Q When you say hard facing--

A Nickel boride, or nickel boride iron combination which they use for spray on, and then heat, and finish.

Q Are they self-bonding in the asprayed condition?

A No. They are self-bonding as soon as the heat is applied to them.

Q After they are fused or welded to the surface?

A That's right.

Q Are you familiar with the--

A They are heat treated afterwards.

Q They are actually--

A Sprayed on and then heat treated.

Q So as to fuse the coating?

A Yes.

Q And weld it to the surface.

A That's right, and then we are also

* * *

A Yes, they do.

Q You had--

A I misunderstood.

Q I was trying to establish whether you had familiarity with the language itself, spray welding.

A Yes, I do.

Q As applied to this process in which the material is placed on the surface by flame spraying and subsequently fused or welded.

A That's right.

Q And that's generally self-fluxing nickel boride containing alloys?

A That's right.

Q Mr. Cook, you testified that in 1963, you received the letter from the Swedish company, concerning a self-bonding material, as depicted in Exhibit 83- A is that correct?

A That's correct.

Q And that material is referred to in Exhibit 83A as Metco 404?

A Yes, that's it. August 20, 1963.

Q That is when you first learned of the Metco 404 material?

* * *

515 patent?

THE WITNESS: No, I don't know about that.

Q Did you ever get an opinion on that patent from your own attorneys?

A No.

Q Did you ever bring that patent to your attorneys' attention?

A I think so. We bring any patent to their attention.

Q Did you ever make an inquiry--

A We had no reason to use the patent, so we wouldn't treat it differently than from anything else. It wasn't a machine patent, so if it isn't a machine patent, our interest is secondary. We look at it this way: This is one material in a whole family of materials used all of which we buy on the outside. It isn't that important. It's not a vital issue about the business, it doesn't hurt our business if we have it or don't have it.

Q But you decided you would like it.

A That's right.

Q Did you ever consider the fact whether this patent might present a problem to your marketing such material?

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# EXCERPTS FROM DEPOSITION OF GEORGE YURASKO

* * *

Q Do you know if after the powder was flame-sprayed or welded on the substrate, referring to Spram?

A I know in some cases it was but whether it was in others, I don't know.

Q Did you have any part,when you were at Eutectic,in developing any materials or anything that was ultimately used in the RotoTec process?

A The ExoTec powder.

Q You had a part in developing the ExoTec powder?

A Yes.

Q Would you tell us your earliest recollection of your involvement in developing the ExoTec powder?

A I was on a trip with Mr. Kapralos, going to Connecticut to survey a potential vendor.

Q Who was this vendor?

MR. GORDON: Is this relevant?

MR. SPRUNG: It may well be.

MR. GORDON: Is this information considered confidential information of the company, who your vendors are?

THE WITNESS: It might be.

MR. GORDON: Actually I see no relevance here at all. I would rather not have him answer.

I instruct the witness not to answer that particular portion.

A During this trip Mr. Kapralos asked me if I thought it was possible to take nickel, nickel base powder and aluminum powder and make some sort of agglomerate and I flatly told him, "Yes, I thought it would be a very simple matter."

Q Did he explain to you why he wanted to make this agglomerate at that time?

A I don't recall really all the details of that, but I do recall that I considered it kind of a simple technique to do this.

Q Did you have any knowledge, prior to that, as to any nickel aluminum composite powders?

A No.

Q You had no knowledge and no knowledge in the field?

A No.

Q And he just asked you simply "Could you make a composite of these two materials?"

A That's right.

MR. GORDON: He said agglomerates.

Is that the word you used?

THE WITNESS: I used the word "agglomerates."

MR. GORDON: I'm sure there isn't any meaning to it but --

Q Do you recall what Mr. Kapralos called it, an agglomerate, composite, or what?

A I don't really recall.

Q Did he explain to you what form he envisioned this powder to be in?

MR. GORDON: What form?

MR. SPRUNG: Yes.

MR. GORDON: Physical form?

MR. SPRUNG: Physical form.

MR. GORDON: Do you understand the question?

THE WITNESS: I understand the question.

A I don't really recall anything specific, you know, about the form at that time. I don't know whether he referred to it as an agglomerate or not but it was a term familiar to me.

Q He just said, in effect, can you put

nickel and aluminum together with single particles, having both of these --

A Right.

Q He made no suggestion of particle size or what it was to be used for?

A No.

Q Did he say it would be the size of marbles, very fine mesh, or anything else?

A No, I don't think he did.

Q Did he give you any indication as to the amounts of the nickel and the amounts of aluminum he might want in the particles, the relative proportions?

A I'm not certain that he did or not.

Q And this occurred during this trip?

A That's correct.

Q Did you discuss this in any detail or was it just a comment, whether you can make this?

A Well, it is only,I think he said he would like to see this powder. What the reasons at that time were, I don't know. And I felt confident that it could be done based on work I had done in the past.

In fact, that was about the extent of it.

Q And this was about what period of time?

A I don't remember the exact dates on that.

Q You have no rough recollection?

A No.

Q You can't place it from about the time you left the company, what year it was in?

A I don't actually recall any specific dates. I just considered it another problem and hit it. I'm sure the records will show the dates on it.

Q After this conversation on the trip, what occurred next?

MR. GORDON: With respect to what?

MR. SPRUNG: With respect to the subject matter.

A When we came back from the trip, I accumulated some materials, and I guess most of them were on hand --

Q When you say you accumulated material, are you talking about physical material, literature, or what?

A Physical material, some of the ~~iron~~ nickel base alloys, aluminum and resins, and I proceeded to

make a small mix, so to speak, to prepare some samples.

Q You had no idea what it was to be used for?

MR. GORDON: At this time?

Q As I understand your testimony, Mr. Kapralos merely said he would like to see this type of powder, he gave you no indication of proportion, size or anything, and that was the substance of your communication, and then you went back and started to work on it; is that correct?

A Yes.

Q So you had no idea what size powder he wanted or what purpose it was to be for, is that correct?

A I could only assume that it was meant to be a spray powder of some type.

Q Why did you assume that?

A We were in that business.

Q Weren't you in other businesses? Weren't you making welding rods?

Did you know this powder wasn't to be put together to make a welding rod?

A I didn't know that.

Q Did you inow it was going to be a welding powder?

A I had to assume that much because Eutectic was in that business.

Q What I am trying to establish --

A I didn't have anything to do, you see, with the electrode end. My sole function there was to make the powders.

Q The Eutalloy?

A And/or maybe anything else they needed it for.

Q What else did they make powders for at that time?

A Well, they were used in electrode formulations, in coating, and they did certain other types of -- they used many other types of powder; tubular wire, I suppose was made at -

Q You just assumed it would be for one of the uses?

A An in-plant use, right.

Q But you had no idea which one?

A Not at that time.

Q When you went about selecting the materials, how did you go about selecting the

particle sizes or the proportions?

A I had done a lot of work in the past with coating powders for electronic applications.

Q Which type of powder?

A Metallic powders, iron-nickel alloys, plain alloys, very fine carbonyl powders, fine, and I had also worked on coloring iron powders, using a coarse mesh, utilizing very fine pigments to coat the surface of these powders, that ultimately were used in magnetic inspection work to afford a degree of contrast.

From this I knew about what size I wanted to use.

Q Your experience was to make electrodes of the same particle size as those used in the Eutalloy process and the same particle sizes in making these electronic components, in your experience?

A Yes. They varied all over the map. Some were coarse and some fine.

Q Which did you decide to make, a coarse powder, a fine powder, or what?

A I used basically what we made there which was minus 140 mesh powder to start with.

Q But you had nothing envisioned in mind what it was going to be used for, is that correct?

A Not at that time.

Q Physically how did you go about making the powder?

A I located a variable speed motor, cut the bottom off of a gallon can and conducted the early tests there.

Q When you say conducted the early tests, what early tests?

A I don't know how many samples I made there, but this thing revolved, I added the nickel, the resin and the pigment and depending on the amount of resin, you could either form an agglomerate or a uniformly-coated powder.

Q What powder components did you use?

MR. GORDON: You said you had a nickel resin pigment? Is that a mistake?

THE WITNESS: In this case it was aluminum.

Q What size aluminum powder did you take, do you recall?

A I think it was about five microns.

Q Very fine?

A Very fine.

Q What else did you take?

A I took a nickel base powder that we had on hand at that time. Later on I located some nickel which was used in electrodes.

Q What size was the nickel?

A Offhand, I would say minus 140 mesh.

Q Coarser than the aluminum?

A Much coarser, yes.

Q Did you envision you were going to coat the nickel with the aluminum?

A Yes, depending on the quantity of resin. As I said, you can either form an agglomerate or a coating.

Q What gave you the idea of coating the nickel with the aluminum? Why didn't you want to coat aluminum with the nickel, coarse aluminum and fine nickel?

A I don't really know but I had these materials available.

Q Did Mr. Kapralos suggest to you that you might want to coat the nickel with the aluminum?

A I don't know.

Q You have no recollection why you did it?

Did you ever try coating the aluminum with the nickel?

A No.

Q Did you ever try using a coarser aluminum powder than nickel powder?

A No.

Q Did you ever try using equal size powders?

A No.

Q What proportions of aluminum did you use to nickel?

A Well, to start with, the percentage was quite high.

Q Of which?

A Aluminum.

Q About what percentage of aluminum to nickel?

A From pigmentation work I knew I could get 17% ~~aluminum~~, of pigment; on iron powder, and I started at a high level.

Q About 17%?

A In that vicinity.

Q Did you ever consider using equal amounts of aluminum and nickel?

A No.

Q Can you tell me why you considered using less aluminum than nickel?

A Only because of this pigmentation work I had done before where it was 17% or 17-1/2% as I recall and then you had an excess of free pigment.

Q Couldn't you have made a fine nickel and a coarse aluminum and put 17% nickel on the aluminum? Wouldn't that have been a possibility?

A It is a possibility but I didn't have such materials available.

It could be done.

Q Do you know if Mr. Kapralos might have suggested that you would want a certain proportion of nickel to aluminum?

A I don't recollect that he gave me any percentages at all.

Q What type of resin did you use?

A At first I think I had a phenolic resin there that was used in the Eutalloy process. I don't recall the name of it. But in the Eutalloy process we put a flux coating on a product named EutaLite, whichis basically the same process.

Q You don't remember what percent resin

you used?

A No, I don't remember exactly what percentage we used there.

Q Would you tell me how you proceeded in the operation? Did you just throw everything together in this drum or did you follow any sequence?

A The sequence in the EutaLite operation was to take the base powder which was coarse, add the resin and then the flux.

I don't recall the specific time intervals involved but the resin was put on the powder, then the fine flux coating was put on and it of course bound to the resin-coated particles, and then it was taken up to the curage stage to set the resin. Then you ended up with basically the same particle size that you went in with.

Q Will you describe the procedure you used in making these initial batches of powder step by step?

A As I recall, when you--

Q Let's start with the drum and you have your materials. Tell us step by step how you went around putting the materials in the drum and --

A Are you speaking of the initial --

Q Yes. You said you got a variable speed motor and a drum.

A Right. Of course, this came from the fact that we also had a agglomerating operation and I decided to make a small revolving disc, put the base ingredient in, diluted --

Q What do you consider the basic ingredient?

A Nickel base powders or nickel oxides, whatever. The base ingredient --

Q What did Mr. Kapralos suggest to you? I thought you testified he said nickel and aluminum.

A He spoke of nickel or nickel base alloy and aluminum.

Q Nickel or nickel base alloy and aluminum?

A Yes.

Q So you tried both?

A Yes.

Q Did you make all the same? You put the nickel or nickel base alloy in the mixing apparatus?

A Yes.

Q Then you added the resin?

A That's correct. I added the resin, and revolved it for a certain length of time to uniformly coat the base material with resin.

Then the aluminum, in the final step, to coat these particles.

Q Then what did you do after it was coated?

A After it was coated I put it in an oven and cured it to set the resin, screened it, submitted it to the technical services area.

Q What did you screen it to?

A Basically minus 140 mesh.

Q Why did you pick that particle size?

A It was a common particle size for us.

Q Then you submitted the powder to Technical Services?

A That's right.

Q Did you just say: Here is a powder?

A I usually wrote a memo and sent it to John Broderick, explaining to him that I had these samples on test and then he would take it from there.

Q Did you suggest what they were to be tested for, making an electrode or what?

A Well, he knew it was to be a spray powder.

Q How did he know it was to be a spray powder?

As I understood your testimony,

Mr. Kapralos told you nothing of what it was to be considered for?

A Well, I don't know what he had on his mind for these powders.

Q But you just gave it to Mr. Broderick and said, "Test it, whatever you can find to test it for"?

A I wasn't aware of all that was going on there, you know.

Q Someone else gave instructions to Mr. Broderick to test it, what it was to be tested for?

A I don't know.

Q But you didn't? You didn't know what the powder was to be used for or tested for, is that correct?

A At that time I'm not so sure. Later on --

Q Let's get to this time. You had only this one conversation with Mr. Kapralos and you made the powder and you gave it to Mr. Broderick, is that correct, or did you have any other discussions with Mr. Kapralos?

A I don't recall any at that time.

Q He just said: Can you make powder

containing nickel and aluminum or nickel base alloy and aluminum, and you said yes, and you went back and made powder and gave it to Mr. Broderick?

A Yes.

Q And told him to test it?

A Yes.

Q Did you have anything in mind when you gave it to him to test it?

A I'm not sure of the sequence of events, really, without having access to records.

Q Let's exhaust your recollection and then we will get into the records.

To the best of your recollection, that is what occurred?

A That is the best of my recollection.

Q Then what happened? Did Mr. Broderick test it?

A Yes.

Q Did you get the test reports?

A Yes.

Q What did the test reports tell you?

A Some were good, some were bad.

Q For what?

A For spraying.

Q For what purpose? For use in the Eutalloy spraying process, use in the Spram spraying process?

A It was either Spram or RotoTec.

Q Did you have RotoTec at that time?

A I don't know but they were playing around with different systems.

Q I am really trying to understand how you gave powders to someone to test without knowing what it was used for and get back test results and see if it was satisfactory.

A Like I said, I don't recall the exact sequence of events. Later on, of course, I knew what it was going to be used for.

Q What was your understanding later on what it was to be used for?

A Well, it became apparent it was going to be a spray powder.

Q For what purpose?

A For a bonding application.

Q At the time Mr. Kapralos first asked you to see if you could put nickel and aluminum together, did you knowwhat a bonding operation was in the spray process?

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if that's what you had in mind?

A Yes.

Q But you certainly wouldn't ask it as a bonding material; would you have expected that he would test it in the normal flame-spray processes which were being marketed by Eutectic at that time?

A I don't know how he tested it.

Q Would you expect this would either be used in the Eutalloy or Spram process, or did you envision some different use?

A I don't know what they were considering. I manufactured the powder. What end uses they were actually considering, you know, I don't know.

Q Mr. Kapralos didn't confide in you what end uses he had in mind or what purposes he had in mind?

A Not in the early stages that we are talking about.

Q Do you recall if Mr. Kapralos offered you a bonus if you would make this?

A Yes, he gave me a bonus for doing this in a rather short length of time.

Q Did that seem unusual to you?

A Well, I don't know whether it was unusual or not.

Q I am talking about to you.

A It was outside the scope of my normal operations which is, I assumed, why I got this bonus.

Q Whom did you report to at that time?

A Mr. Quaas.

Q Did you usually do work Mr. Kapralos instructed you or authorized you to do?

A No, except I think Mr. Quaas was out of the country at that time.

Q You felt you were reporting to Mr. Kapralos in Mr. Quaas' absence?

A I didn't feel that I was reporting to him. I just felt it was a challenge.

Q I thought you said it was something that could be done very simply and no problem.

A It was done simply.

Q Why did you consider it a challenge?

A Just to prove it could be done.

Q Did you have to prove to yourself it could be done or did you think it was something that was pretty obvious and pretty simple to do?

A Well, he wanted the powder and based on

my past experience I thought it was simple to do this.

Q So it wasn't a challenge to you, really?

A Well, here again it depends how you interpret it.

Q How did you interpret it?

A I wanted to prove to him that it could be done.

Q Were you ever offered a bonus at Eutectic for doing any other work at the company?

A None other than the usual bonuses.

Q This was the first time someone said to you: If you can make this material, we will give you a bonus?

A That's the first time.

Q How much of a bonus did they offer you?

A It was either $200 or $400, I'm not certain it was.

Q Is my understanding correct, he said: Make a nickel and aluminum powder with the particles of nickel and aluminum stuck together, is that correct, and I'll give you a $200 or $400 bonus, you had no knowledge what it was to be used for, what the composition and what the particle size was?

A At that time, no.

Q You proceeded to make a nickel, and nickel-base alloy powder coated with up to about 17% aluminum and gave it to Mr. Broderick to test for certain specific purposes, is that correct?

A Yes.

Q I still cannot understand what you had in mind when you gave it to Mr. Broderick to test, or what instructions you gave Mr. Broderick.

MR. GORDON: Is there a question?

Q What did you envision that Mr. Broderick would do with this powder when you gave it to him to test?

A At that time I don't really know. There were many things going on there in the R & D area that I wasn't aware of.

Q The nature and type of tests were not your suggestion?

A No.

Q Mr. Yurasko, at the time that Mr. Kapralos asked you to make this nickel-aluminum powder, did he explain to you that Metco had such a powder on the market?

A I don't recall anything to that effect.

Q Do you know that Metco has manufactured such a powder?

MR. GORDON: As of the present time?

MR. SPRUNG: Yes.

A Later on I got to see a patent.

Q That was the first time you learned of Metco?

A I heard the name prior to that.

Q I mean Metco's involvement in the nickel-aluminum field?

A Yes.

Q About how much later was that?

A I don't know. I don't recall how much later. It was after the initial testing.

Q I hand you a document, Defendant's Exhibit 2, and ask you if you can identify that document.

A It certainly looks like my printing.

Q Can you identify that at all?

A Yes. These apparently are samples that I had prepared, 160--1,2,3 to 6, indicating the base materials, in fact all the ingredients.

Q Do those reflect the first initial samples that you made in accordance with the request

by Mr. Kapralos and the bonus given?

A Yes.

Q And you gave these sample numbers 160-1,2,3,4,5,6.

Do you recall why you picked those numbers?

A I picked those numbers because the 160 denotes the department number which I operated, and the 1 through 6, the sample numbers.

Q As you made the sample numbers you numbered them 160-1,2,3,4,5,6 in sequence?

A Yes.

Q Did you make subsequent samples after that?

A I probably did.

Q Did you number them with subsequent numbers?

A They have have the same numbering sequence.

Q I hand you a document marked Defendant's Exhibit 12 for identification.

Can you identify that document?

A I don't see that I signed that memo but --

Q Are those your initials as you normally

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is that correct?

A Yes.

Q So that these samples were completed as of that date?

A The samples indicated here, yes.

Q And at that time, you knew something about Metco Ni-Al?

A A little bit.

Q Does this document refresh your recollection as to what you knew about Metco Ni-Al, at that time?

A Well, I knew it was to be a spray powder at this time. I probably didn't know the exact sizes that were involved, but that it was to be used at least, in part, for a spray application.

Q I understood you testified that the first knowledge you received of the Metco nickel-aluminum material was from the Metco patent, is that correct?

A Probably so.

Q So you were probably shown the patent before this date?

A Or thereabouts, right.

Q And you would have read the patent?

MR. GORDON: Did he read it?

Q Did you read the Metco patent when you were shown it?

A I went over it briefly. Either Mr. Quaas or Broderick had it, I believe, I'm not sure.

Q Not Mr. Kapralos?

A I really don't know who had it.

Q You saw the patent, you probably saw the patent and went over it prior to 3-23-70, when you submitted these samples?

A Yes. (Possibly)

Q It says, "Should initial tests on these show promise we should submit samples for metallographic examination and be prepared to conduct bond strength test versus the Metco counterpart."

A Right.

Q Would that indicate to you that the Metco material was used as a bonding material?

A Yes, it would.

Q And that this material was intended as a bonding material?

A Yes, it would.

Q Is it possible, then, you had the Metco

patent at the time you made these samples?

A I didn't see that patent until after I had made these initial samples.

Q How much time elapsed between the time you made the samples and the time you submitted it to Mr. Broderick?

A What is the date on that?

Q 3-23-70.

MR. GORDON: Are you referring to Exhibit 2?

THE WITNESS: Yes.

MR. GORDON: It's undated. At least we haven't been able to find a date anywhere.

A Probably a couple of days from the time I made them until they were given to Mr. Broderick.

Q Someone would hae given you the Metco patent within those few days?

A I don't recall the exact span of time there.

Q What I am trying to establish, according to the best of your recollection, you received this knowledge of Metco's patent, what these materials were to be used for, between the time you made the samples, 160-1 through 160-6, and the time

you submitted the 160-1 through 160-3 for Mr. Broderick for testing, is that right?

A I'm trying to get a feel for my timing here, from the time I got back, and I don't recall the exact span of time.

Q From Defendant's Exhibit 12 you must have had this knowledge before you submitted it to Mr. Broderick, is that correct?

A I was either told or saw the patent after, you know, I made these initial samples and what the potential end use was.

Q After you made them but before you submitted them to Broderick?

A Yes.

Q There is no possibility you could have been told before or why you were making them?

A No.

Q I hand you a document marked Bates No.50253 and ask you if you can identify that document.

A It is a test report, laboratory particle size test report.

Q Are you familiar with that report at all or can you in any way relate it to the samples, any of the samples 160-1 through 160-6, as depicted in

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Q When you refer to Defendant's Exhibit 100, you say that could be the starting material, which you requested for making the samples?

A Right, that could be.

Q And would that fix the date 3-7-70, would that refresh your recollection as to the period of time between making the materials and submitting them to be tested to Mr. Broderick, as shown on Exhibit 12, 03-23-70?

A Yes, that would have to tie in.

Q Does that refresh your recollection that you probably started work, probably in the beginning of March of 1970, and completed the work around the 20th or 23rd and submitted it to Mr. Broderick?

A Yes.

Q And within that interim, you received knowledge of the Metco patent, Metco material?

A Yes.

Q I would like to hand you Defendant's Exhibit 13 for identification.

Can you identify that document?

A It is a report on a nickel aluminide.

Q What does the 450 mean to you?

A It is the number of the Metco composite.

Q And this sieve analysis was made at 3-12-70?

A That is correct.

Q That was right in the period you were making this powder, is that correct?

A That is correct.

Q Do you know what Metco 450 is, today?

A As I recall, Metco had two of them. I don't remember the numbers any more but one had in the area of 20% aluminum and one around 4-1/2 to 5%.

Q When you made samples 160-1 to 160-6, what percent aluminum to nickel did you use, what range?

A That was 12 to 20.8% by weight.

Q Do you recall from the Metco patent what that range might suggest?

A No.

Q Now that your recollection may be refreshed from Defendant's Exhibit 12, as to what tests you asked Mr. Broderick to perform, do you have any recollection as to what the test results were?

A Well, they are indicated on this report.

MR. GORDON: Referring to Exhibit 2.

A Wherein it is indicated 160-1, 160-2, 160-3, and sample 160-6, according to his tests were okay.

Q And they were just tested for bond tests, as indicated on Exhibit, Defendant's Exhibit 2?

A Yes, but I don't know what else he might have tested for.

Q Would that indicate to you that was the important feature being tested?

A Yes.

Q Do you have any recollection that that was the purpose of this material, as a self-bonding material? Does this refresh your recollection?

MR. GORDON: This is the first time you have used the word "self-bonding."

MR. SPRUNG: Yes.

MR. GORDON: I'm not sure that the witness is familiar with that.

MR. SPRUNG: We have used it before in this testimony.

Do you want it read back?

MR. GORDON: I don't recall but I'll have to take your word for it.

* * *

MR. GORDON: I don't recall that.

Will you repeat the question?

(Question read.)

MR. GORDON: From looking at these two exhibits can you answer that question?

A Yes, that is the bond, yes.

Q You made these initial samples. Did you make any further samples?

A I probably did.

Q Do you have any recollection of making any further samples?

A I'm sure I did but I don't know how many.

Q Exhibit 13 indicates as of 3-12-1970 you had an analysis of the Metco 450 nickel aluminide? That is, at Eutectic there was an analysis of 450 nickel aluminide?

A Yes.

Q Do you recall whether at any point you attempted to duplicate that material?

MR. GORDON: He personally?

Q You personally.

A Not up to this point.

Q Let me ask you, at your time at Eutectic, did you ever try to duplicate the Metco 450 material?

A Well, later on we got a specification, an aircraft specification from somebody in the field, and it spelled out pretty much the specs required, and in that spec they tied down the particle size, the aluminum level, plus some other residual things.

Q And you tried duplicating that?

A Then we tried to prepare material to meet that spec.

Q And that was the same as the Metco, to your knowledge, 450 material?

MR. GORDON: There is a time problem. At the time you got the spec did you know it was the same as --

A I don't know.

Q You knew as of 3-12-70 of the Metco 450 material?

MR. GORDON: He personally?

Q I am talking about you personally.

A I don't know. what appears to be Mr. Quaas' initials are on that and I don't know whether I got a copy of that.

Q Do you have any recollection?

A No.

Q What is the earliest recollection you have of knowledge of the composition of the Metco 450 material?

A I think when I saw the aircraft specification.

Q At that time?

A Yes.

Q I thought you testified at this time Mr. Quaas was out of the country?

A He may have been.

Q Could you explain how his initials would appear on this?

I am referring to Exhibit 13.

A I don't know if he was out of the country here on this date or not, but at the time possibly that Kapralos and I were taking this trip, he was out of the states.

Q He may have come back in the interim?

A Yes.

Q I hand you Defendant's Exhibit 17 for identification.

Is this the specification you were referring to?

A It looks like it.

(Luncheon recess: 12:00 Noon.)

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that's when I made it.

Q I hand you a document which has been marked Bates No. 50250 and ask you if you can identify it.

A It is a purchase requisition made up by me.

Q What was it for? Did it have any relationship to your initial work leading to the ExoTec material?

A Probably I wanted to order more nickel oxide to prepare powder from, and some carbonyl nickel, which I also probably wanted to use.

Q Did you make powders other than that depicted in Exhibit 2, which contained nickel oxide as a core, as opposed to nickel, or a nickel base alloy?

A I don't remember.

Q Do you recall any work being done on core materials containing nickel oxide or coated with nickel oxide in an attempt to avoid the Metco patent?

A I believe I oxidized some nickel and coated it with aluminum, to show that the reaction was basically oxygen with aluminum.

* * *

laboratory to Mr. Joseph, indicating that a micro-analysis is required in samples 160-1, 2, 3 and 6.

Q The term "pre-bond alloys"--

MR. GORDON: Have you established if he has seen it before? His name does not appear on it.

Q Are you familiar with that document?

A I believe this is the first time I have seen it.

Q Do you have any knowledge of the term "pre-bond alloys" being used at Eutectic during this development period in the beginning of 1970, March and April 1970?

A No.

Q You never heard that?

A I don't recall haring that term.

Q I hand you a document marked 50228 and ask you if you have seen that document before?

A I don't remember it specifically.

Q Whose handwriting is that?

A I can't identify it.

Q I hand you document marked Defendant's Exhibit 59.

Are you familiar with that?

A This is a memorandum I wrote to Mr. Quaas describing the early procedure.

Q Used in makin samples 160-1 to 160-6?

A Yes.

Q I hand you a document and ask you if you can identify the writing on that.

A It doesn't look like my printing.

Q Have you ever seen that document? Can you identify it in any way?

A I can't say that I have seen that before.

Q Can you identify it?

A Well, it is the procedure used for preparing nickel aluminum powder.

Q I hand you a group of documents and ask you if you can identify the writing on any of them.

A I don't know whose writing that is. These look like some of my scribbling notes here (indicating).

MR. SPRUNG: I ask the reporter to mark three documents, 50134, 50126 and 50128.

xx (Documents 50134, 50126 and 50128 marked Defendant's Exhibits 103, 104,105 for identification.)

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oxide in between the aluminum and the nickel?

A Yes.

Q And that is shown in the drawing on the left-hand side, marked "Eutectic"?

A Yes.

Q Do you know if Eutectic ultimately commercialized a product having nickel, metal and aluminum or whether they made a product, nickel oxide and aluminum?

A When I left it was nickel and aluminum and I don't know what has been commercialized by the oxide route.

Q You have no knowledge of commercialization of the oxide but you do have knowledge of commercialization of the aluminum with the nickel, is that correct?

A Yes.

Q Do you know who Pete Constantine was?

A Pete Constantine was in my employ, having worked for Metco. I don't recall the exact dates that he worked for me, but I had to leave him go. I let him go. He worked there for a short period of time.

Q Was he at Eutectic at the time you initially

made samples 160-1 through 160-6?

A I can't really say. I don't remember his hiring.

Q He did appear around April 15, 1970, at the time you wrote Defendant's Exhibit 20, is that correct.

A Yes.

Q Do you know if Pete Constantine had any knowledge of Metco's 404 and 450, nickel aluminide?

A A little.

Q Did he ever discuss their product with you at that time?

A We probably discussed it off and on. I can't say tha he really knew how it was made. He knew a little bit more about testing, I believe.

Q But he was familiar with the products?

MR. GORDON: The existence?

Q Was he familiar to your knowledge, of their composition?

A I don't know that I revealed the composition to him.

Q I am talking about the Metco materials.

A He probably did.

Q Did you do any other work in an attempt

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area by the technicians there.

Q Did you observe those tests or have any part of them?

A I don't recall observing the tests, only reporting the results as reported to me.

Q Metco 450 was also tested?

A Yes.

Q Who is Mr. Doody?

A Mr. Doody used to be a vice president there.

Q Of Eutectic?

A Yes.

Q What was his part? Did he have any part in this development?

A I recall none.

Q I hand you Defendant's Exhibit 46.

A Mr. Doody was in the sales area and apparently requested samples, which we submitted.

Q What does the term "ExoTec-Therm mean to you?

MR. GORDON: That says "Eutec-Therm."

Q I'm sorry, Eutec-Therm.

A Probably a trade name they were going to apply to this material.

Q Do you know if they ever applied it?

A Not to my knowledge.

Q During the period you were working, developing these powders that developed into the ExoTec material, how long did Mr. Constantine work with you?

A I think it was a little over a year although I am not certain.

Q Pretty much from the beginning of the development as reflected by this April date, until the following year in April?

MR. GORDON: The testimony was March as I recall.

MR. SPRUNG: The first date we have Constantine's name was in April.

A I don't recall exactly how long he worked, a little over a year, I think.

Q Who else was involved in this development? Can you tell me what Tanzman's role was?

A Tanzman was in the research department, and whether he did any work there or not, I don't know. My contacts with him were very limited, practically none.

Q What was the period he was there, do you

recall?

A It seems to me he left to go to Metco and then he came back for a brief period. I had very little contact with Mr. Tanzman.

Q And you can't give us the dates?

A No, I can't.

Q How about Mr. Patel, what was his involvement in this?

MR. GORDON: Meaning the development of Exo-Tec?

MR. SPRUNG: Yes.

A I don't think he was there when we developed this.

Q When was your first contact with him?

A Here again, he was brought into research and we were very remote in our work areas.

As I understood it, he was going to go to India. What-all he worked on, I don't really know.

Q Was it your practice in making these various samples to evaluate them against the Metco material and see which compared most favorably with it?

MR. GORDON: Personally?

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MR. SPRUNG: I would ask the reporter to mark this Exhibit 110, No. 50194.

xx (Document No. 50194 marked Defendant's Exhibit 110 for identification.)

A This is a memo from me to Broderick.

Q Concerning this development work on the materials that ultimately led to the ExoTec powder?

A In connection with ExoTec.

Q Does that refer to Mr. Tanzman?

A Yes. He recommended some kind of a test there.

Q What type of test did he recommend?

A I think they overlaid, sprayed aluminum and then another coating, another material on top of that.

Q Doyou know if there were any reports on Mr. Tanzman's recommendation other than this hand-written note?

A I don't know.

Q Do you recall any other recommendations that Tanzman may have made concerning this development?

A None to me.

Q The purpose of all this development work

was to develop a flame spray nickel aluminum bonding material that would meet the aircraft specifications that you previously testified to?

A That is what we were striving for.

Q That would be equivalent to the Metco 450?

A Apparently, yes.

Q Did you ultimately reach that goal?

A We prepared the spec.

Q Were you attempting to make a material that was equivalent to Metco's 450?

A We were making the material to meet the PWA spec.

Q Were you attempting to make a material that was equivalent to Metco's 450?

MR. GORDON: He just answered that question.

Q Yes or no.

MR. GORDON: He answered the question.

Q Yes or no?

A Apparently the Metco material falls within that spec.

Q Did you have Metco material 450 on hand and making comparison tests of --

A Yes, comparison tests.

Q Of your material against Metco 450?

A Yes.

Q And you were trying to make a material that was at least equivalent to the Metco 450?

A And to meet the PWA spec, yes.

Q And were you successful in making such a commercial material?

A Yes.

Q What was that material ultimately marketed, under what designation, do you know?

A I would say ExoTec.

When was this development completed, do you recall?

A I don't recall.

MR. SPRUNG: I will ask the reporter to mark this document, Document No. 50312.

XX (Document No. 50312 marked Defendant's Exhibit 111 for identification.)

Q Does that document refresh your recollection as about the time the powder had been developed to a point of commercialization?

A Yes.

Q Doyou know how long after this memo, as depicted in Defendant's Exhibit 11, you were able to

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# EXCERPTS FROM DEPOSITION OF MAHESH S. PATEL

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Q What were your duties at Metco?

A My duties was to follow my project engineer.

Q Who was your project engineer?

A My project engineer was Mr. F.N. Longo.

Q And you worked under his supervision?

A Right.

Q Were you familiar with any composite powders being manufactured, being worked on at Metco during this period of employ?

A Manufacturing?

Q Being worked on, developed or manufactured, composite powders containing nickel and aluminum components.

A No.

Q You are not familiar with any?

A No.

Q You were not familiar with Metco 450 powder?

A No.

MR. KAPRALOS: Excuse me, one second. What do you mean by familiar? Was he aware of it, did he work on it?

Q You had no knowledge--

A I had no knowledge when I was there.

MR. GORDON: You are asking as to his period of employment?

MR. SPRUNG: Yes.

Q Did you do any work on the formulation of flame spray powders?

A Yes.

Q Did you do any work on the formulation of flame spray composite powders? Do you know what I mean by a composite powder?

A No.

Q You don't know what I mean or you didn't do any work?

A I am saying no to that I worked— on any composite powder?

Q Do you understand what I mean when I say composite powders? Do you understand what the term means?

A Composite powder?

Q Yes.

A No.

Q Do you understand what I mean when I say clad powders?

A Right.

Q What is your understanding of the term clad powders?

A Something sticky on another thing, something sticking together.

Q Are you familiar with any work on powders having a core of one material with a cladding or coating?

A That I understand.

Q You were.

MR. GORDON: He said he understands. Now ask your question.

Q You understand what that is. Did you do any work on that type of powder when you were at Metco?

A Yes.

Q Would you describe the powders you worked on?

A I worked on abradable powders and Metco 301 is powder like that.

Q What do you mean by abradable powder?

A That's exactly what I mean, abradable powder.

Q What was the composition of the powder?

A I don't recall.

Q Are you familiar with a process by which

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letter.

Q Do you know if a copy of that letter is available?

A I might have it, I don't know.

MR. SPRUNG: Would you produce it?

MR. KAPRALOS: Yes.

Q What were your first duties at Eutectic? In what department did you go to work at Eutectic, your first assignment.

A What department?

Q Yes.

A Research and development.

Q Whom did you work under?

A Under Mr. Quaas.

Q Did you directly report to Mr. Quaas or was there--

A Directly to Mr. Quaas.

Q Do you recall in what field your first work was?

MR. GORDON: Would you qualify field?

Q What did you do? What was your first research task?

A My first task was to familiarize myself with the materials.

Q That were being manufactured by Eutectic?

A That were being manufactured by Eutectic.

Q Among the materials that you familiarized yourself with, was there anything involving nickel aluminum combinations?

A No.

Q Were there any clad powders?

A No.

Q No clad powders whatsoever?

A No clad powders whatsoever, in any form.

Q Did you familiarize yourself with all the products that Eutectic was manufacturing at that time or developing?

A I tried to familiarize myself with their existing product which was related to electrodes and flux.

Q Just electrodes and fluxes?

A Yes.

Q How long did you spend at Eutectic trying to initially familiarize yourself with the products they were manufacturing?

A Almost a year.

Q And you did nothing else during that year?

A I did nothing else.

Q What did you do, just read their brochures or how did you go about familiarizing yourself with their products?

A They have many raw materials and other materials related to electrodes and flux, which takes a long time to familiarize.

Q Did you physically get samples?

A The particle size, screening, chemistry, things like that.

Q I still don't quite understand. Did you go through a catalog that they had?

A I went to Q.C., to see how they go about with the powders, familiarizing myself.

Q In other words, just learning the business.

A Yes.

Q In learning the business did you also become concerned with the materials they were developing at that time or only the materials they were manufacturing?

A Manufacturing.

Q You were only concerned with manufacturing?

A Not manufacturing. Only products related to electrodes.

Q Only materials related to electrodes?

A And not the mode of manufacturing.

Q You came to Eutectic at the end of '70?

A Beginning of '71 or end of '70, last days of '70.

Q And you spent until 1971, approximately, only familiarizing yourself with the composition of the electrodes that Eutectic was working on, is that correct, and fluxes?

A Mainly raw materials.

Q What do you mean by raw materials?

A Like electrodes has its own composition and I wasn't familiar with that.

Q What were you familiar with?

A The materials going into the electrodes.

Q In other words, the materials that Eutectic used to manufacture the electrodes with, is that correct?

A Say it again, please?

(Question read.)

A That's correct.

Q Are you presently familiar with a product

manufactured by Eutectic known as ExoTex?

A Presently?

Q Yes.

A Yes.

Q When did you first acquire any information concerning this type of material?

A When?

Q When did you first acquire information concerning this type of material, and I am referring to the ExoTex?

A You want a date?

Q Approximately.

A Maybe the end of '71 or ~~something~~ around that time.

Q The end of '71?

A Yes.

Q And how did you first acquire this information?

A When I was assigned coloring the project, the ExoTex material.

Q The end of '71?

A Yes.

Q And how did you first acquire this information?

A When I was assigned coloring the project,

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A That it bonds on a substrate?

Q When it is sprayed.

A When it's sprayed.

Q How does that differentiate it from ordinary flame spray material?

A The ordinary flame spray material will not bond, compared to this powder.

Q When did you learn, if you did, what the composition of this ExoTec material was?

A Like I say again, I might have learned along the line of this project what composition was, but I don't remember, or I don't recall anything that I learned right away because I had no necessity to learn what the composition of that powder was. I had no necessity of learning it was a bond coat or used for painting.

Q What is your present understanding of what the ExoTec powder is?

A My present understanding of the ExoTec powder is nickel and aluminum powder.

Q In what form?

A Core material and you have aluminum outside.

Q Nickel core material?

A Nickel core material.

Q And it has an aluminum clading?

A Right.

Q Are you familiar how the clading is applied?

A Yes, like to put in a pot and mix with the mixer.

Q The same way that you clad the powders as described in Exhibit 81?

A Similar matter, right. Let me tell you exactly. The way I made that powder was, put in the beaker and with kitchen type mixer, mix it. I don't know how this powder was made. Maybe in another type of mixer. I don't know.

Q Did you at one time come to the understanding that the powder was made by mixing nickel powder, a resin and finely divided aluminum powder together.

A That's my understanding at the present time.

Q When did you acquire this knowledge?

A As the project progressed, I am beginning to learn about this.

MR. GORDON: Can you pinpoint the date following, how many months or weeks?

THE WITNESS: I might have learned it-- it takes a few days to acquire a this knowledge. It's

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A Which helps any reaction.

Q Assiststhe reaction and doesn't take part in it?

A Doesn't take part in it, but, like I said, I don't exactly understand right now what it exactly means.

Q Before, you mentioned Mr. Carroll as somebody whom you had worked with at Metco, and later at Eutectic. Was there anyone else that you recall working with first at Metco and later Eutectic?

A Not that I recall.

Q You don't recall, you say?

A Not do I recall it.

Q Did you ever come across a fellow named Tanzman?

A Yes.

Q Where did you first meet him?

A I met him at Metco.

Q While you were both working there?

A While I was working at Metco.

Q And he was working there as well?

A He was working at Metco.

Q Did you ever work in connection with him, working in the same tasks, alongside each other or

doing supplemental work?

A No, sir, because every engineer at Metco is assigned different projects. I had no need to work with him.

Q Did you ever consult with him there?

A No, sir.

Q What was your understanding as to the kind of work he did at Metco?

A I don't know what he did work on.

Q You never discussed it?

A I never discussed with him.

Q Did you leave Metco while he was still at Metco?

A This is three years ago. I don't remember whether he left before me or I left before him.

Q But it is your understanding he is no longer at Metco?

A That is what my understanding is.

Q After you came to work at Eutectic, did Mr. Tanzman work there at any time?

A After I left Metco?

Q And joined with Eutectic, did Mr. Tanzman work there at any time?

A I saw him at Eutectic.

Q Was he doing work there or visiting?

A He must be working because I saw him working there.

Q What is the nature of the work that he has been doing at Eutectic, if you know?

A I don't know.

Q Did you ever discuss with Mr. Tanzman your leaving Metco and coming to Eutectic?

A Not that I know of.

Q When you were at Metco, did you ever meet a gentleman by the name of Mr. Constantine?

A Yes, I saw him walking around there, at Metco.

Q Was Mr. Constantine still at Metco at the time you left?

A When I left Metco, I didn't see him working over there.

Q When you joined with Eutectic, was Mr. Constantine at Eutectic at that time?

A I don't recall seeing him over there.

Q Have you ever seen him at Eutectic?

A No.

Q Are you aware of whether or not Mr. Tanzman has done any work at Eutectic in the flame spray field?

A Like I said, I didn't know what he was doing there.

MR. GORDON: Is the question Eutectic or Metco?

MR. SPRUNG: Eutectic.

A No, I didn't know either.

MR. KRAMER: I have nothing else.

MR. SPRUNG: We would like to make another request that you ascertain the employment of Mr. Tanzman in connection with Eutectic.

MR. GORDON: I'm not quite sure what you want.

MR. SPRUNG: I would like to know what projects he worked on, whether he had any relation to the subject matter in suit, whether he is regularly employed, consulting or what, and if he is a consultant, we would like to see his consulting agreement.

MR. KRAMER: At this time, I think we can adjourn the deposition. I can't complete the examination because the Xuper Bond patent application which had been promised to us has not been produced, so that it may be necessary for you to come back on another date.

MR. GORDON: Did you read the application?

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Service of ONE copies of the
within APPENDIX is hereby
admitted this 15th day of
FEB. 1977

Signed Mary H
DEFENDANT-APPELLANT
Attorney for AND CROSS APPELLEE

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